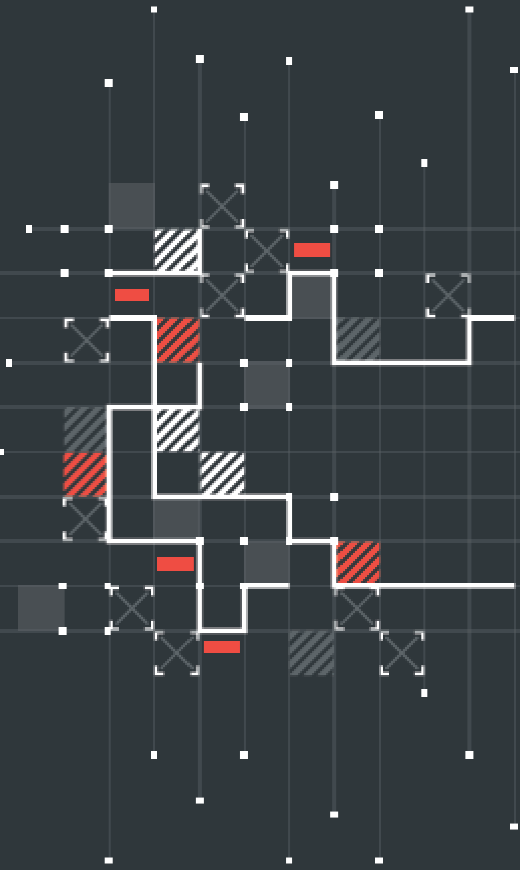

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PREFACE

The rapid transformation in science and technology necessitates a fundamental reconfiguration not only of new products but also of production processes, material design approaches, and sustainability paradigms. Today, multidisciplinary research fields ranging from additive manufacturing technologies to functional chemical materials, and from advanced ceramic systems to waterless cosmetic production, are positioned at the intersection of engineering, materials science, and industrial sustainability.

This book aims to serve as a guiding reference for both the academic community and industrial stakeholders by addressing the scientific foundations and industrial implementations of modern manufacturing technologies from a holistic perspective. Beginning with additive manufacturing and FDM processes, the volume presents in-depth and original contributions on topics such as the development of extreme-temperature-resistant ceramic systems, contemporary scientific strategies for enhancing functional performance in heterostructures, waterless production technologies in the cosmetics sector, zero-waste and carbon-negative process design in the food industry, and process–cost optimization in the manufacture of functional chemical materials.

Moreover, the final chapter, which comparatively examines the influence of substrate effects on the hardness and scratch behavior of CrN-coated systems, constitutes a significant reference for researchers working in the field of surface engineering. Each chapter of the book has been prepared through the original work of domain experts and is intended to establish a strong bridge between current scientific literature and industrial practice.

We hope that this volume will provide both theoretical insight and practical perspective for undergraduate and graduate students, academics, R&D specialists, and industry professionals, and we would like to express our sincere appreciation to all contributing authors and stakeholders who have played a role in the preparation of this work.

Prof. Dr. Mehmet ŞİMŞİR
Prof. Dr. Salih Cem İNAN
Assoc. Prof. Dr. Sayiter YILDIZ
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CHAPTER 1

ADDITIVE MANUFACTURİNG AND FUSED DEPOSITION MODELİNG: PRINCIPLES, PROCESS PARAMETERS, AND MATERIALS

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INTRODUCTION

Today, manufacturing technologies are undergoing a significant transformation process in line with the required performance requirements, product designs with complex geometries, and the increasing need for product development. Although traditional manufacturing methods have formed the basis of industrial production for many years, these methods have certain limitations in terms of design freedom, material usage efficiency, and production flexibility (Gibson, Rosen & Stucker, 2015). These limitations have led to the development and use of new generation production methods.
In this context, Additive Manufacturing (AM) technologies are an innovative production method that enables the conversion of digital design data into final products. Additive manufacturing is the process of obtaining final products by layering material on top of each other based on three-dimensional models designed using computer-aided design (CAD) programs (ISO/ASTM 52900, 2021). Unlike traditional manufacturing methods, additive manufacturing is not based on removing material from a surface, which is its most distinctive feature.

This production method offered by additive manufacturing technologies provides significant advantages, particularly in terms of reducing unnecessary material usage, shortening production time, and enabling the production of complex geometries that are difficult and time-consuming to produce using traditional methods (Ngo et al., 2018). Thanks to these advantages, additive manufacturing is increasingly in demand in many different sectors that require high performance, lightness, and functional design, such as aerospace, defense, biomedical, and automotive. Particularly in defense and aerospace applications, the production of parts with internal channels, voids, and topology optimization has become possible with additive manufacturing technologies (Gibson et al., 2015).

Additive manufacturing methods are classified into different categories based on the energy source used, the type of material, and the production principle. Among these methods, Fused Deposition Modeling (FDM) technology, widely used in thermoplastic-based production, is frequently preferred in both academic studies and industrial applications due to its wide range of materials and ease of process control (Turner & Gold, 2015). The FDM method is based on the principle of extruding molten thermoplastic filament from a nozzle at a specific temperature and layering it on top of each other.

However, the mechanical, thermal, and surface properties of parts produced using FDM technology vary depending on the parameters used in the production process. Parameters such as nozzle temperature, layer thickness, printing speed, infill density, pattern structure, and production direction and angle are among the key factors that determine interlayer bonding, the microstructure of the part, and its behavior (Turner & Gold, 2015; Ngo et al., 2018). Therefore, it is more accurate to consider FDM technology not only as a production method but also as an optimizable production process.

This chapter of the book addresses the concept of manufacturing in a general framework, examines additive manufacturing technologies in comparison with traditional manufacturing methods, and highlights the advantages offered by additive manufacturing. Subsequently, the working principle of FDM technology is detailed; the basic production parameters used in FDM devices and the effects of these parameters on production quality are discussed. Finally, the thermoplastic materials used in the FDM method and their place in engineering applications are evaluated, presenting the position of additive manufacturing within modern manufacturing technologies from a holistic perspective.

MANUFACTURING

Manufacturing is defined as the process of converting raw materials or semi-finished products into functional and economically valuable products through specified physical and chemical processes (Kalpakjian & Schmid, 2014).
This process involves different methods such as:

- Forming,
- Machining,
- Casting, and
- Surface treatments.

The production method to be applied is determined by considering the geometric structure of the part, the type of material, and the intended use.

Traditional manufacturing methods are mostly based on the principle of removing material from the surface. Achieving the desired geometry requires the removal of excess material. This means significant material loss and time-consuming production times, especially for parts with complex geometries (Groover, 2020). In addition, traditional manufacturing methods are widely preferred in many industrial applications today in terms of high dimensional accuracy, surface quality, and mass production capability.

Developments in manufacturing processes aim to make production processes more efficient, flexible, and sustainable. In this sense, the use and proliferation of digital design and computer-aided manufacturing systems have played a crucial role in diversifying production processes (Kalpakjian & Schmid, 2014).

Additive manufacturing technologies, as part of this transformation, present themselves as an alternative production method to traditional manufacturing methods.

Figure 1 shows a general representation of the machining process.

Figure 1: Machining

ADDITIVE MANUFACTURING

Additive manufacturing (AM) is the general term for layered production technologies that convert three-dimensional models created in a computer-aided design (CAD) environment into the final product. In this production approach, the product is created by adding layers of material on top of each other (ISO/ASTM 52900, 2021).

Additive manufacturing technologies have shown significant and rapid development in recent years, particularly due to the design flexibility they offer in the production of parts with complex geometries. Internal channels, lattice structures, and topologically optimized designs can be produced as a single piece using additive manufacturing (Gibson, Rosen & Stucker, 2015). This provides important advantages in terms of reducing part weight and increasing functional performance.

The availability of a digital production infrastructure enables the effective use of additive manufacturing technologies in rapid prototyping and product development processes. The ability to make design changes directly in the digital environment and quickly transfer them to production has been a reason for choosing additive manufacturing, especially in R&D activities (Ngo et al., 2018).

Comparison of Additive Manufacturing and Traditional Manufacturing

Traditional manufacturing methods are generally suitable for mass production and should be preferred. However, these methods require high tooling costs and long setup times for the production of parts with complex geometries (Groover, 2020). Furthermore, depending on the production principle, material waste is a significant disadvantage.

Additive manufacturing, on the other hand, offers advantages in low- and medium-volume production, customized designs tailored to individual needs, and the production of functional parts. The absence of tooling requirements, short pre-production preparation time, and design flexibility are among the prominent aspects of additive manufacturing compared to traditional manufacturing (Gibson et al., 2015). However, it should not be overlooked that traditional production methods still maintain their superiority in terms of production time, surface quality, and certain mechanical properties.

Therefore, today, additive manufacturing technologies are considered a complementary production approach rather than a replacement for traditional manufacturing methods (Gibson, Rosen & Stucker, 2015; ISO/ASTM 52900, 2021).

Advantages of Additive Manufacturing

One of the main advantages offered by additive manufacturing technologies is high design freedom. Geometries that are difficult or impossible to produce using traditional manufacturing methods can be produced without requiring additional processing thanks to additive manufacturing (Ngo et al., 2018). This enables the implementation of innovative designs aimed at improving product performance.

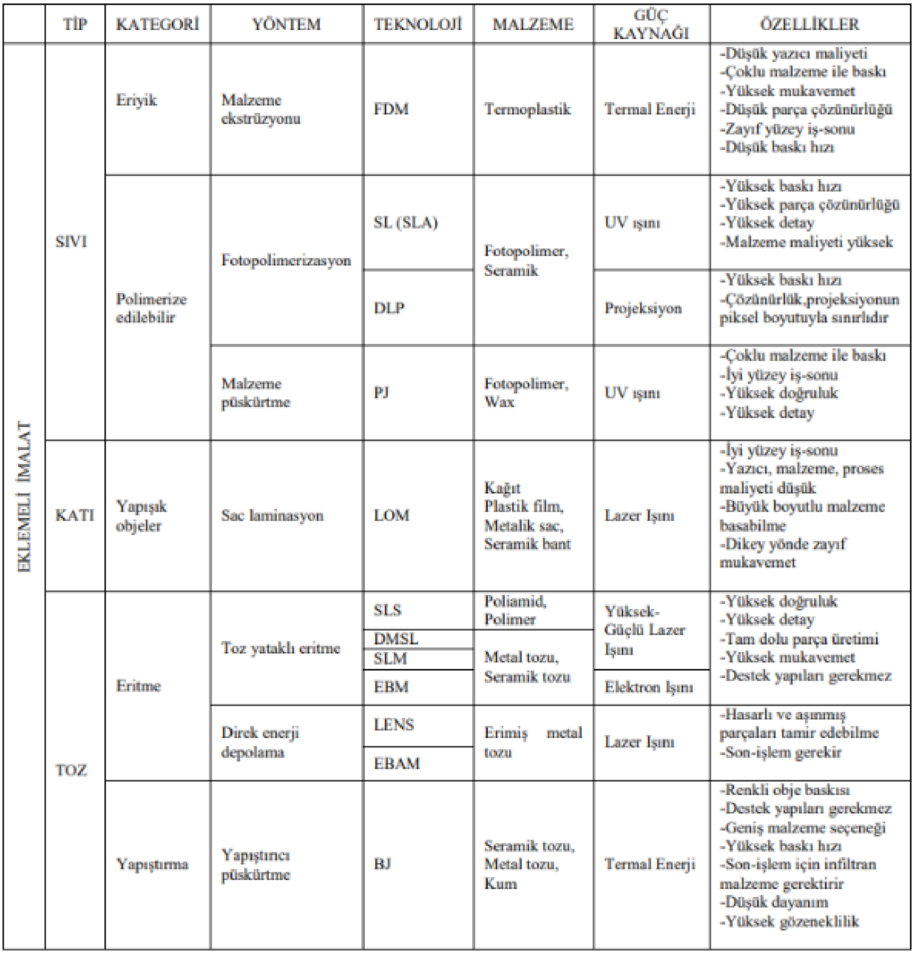
In addition, additive manufacturing offers significant advantages in terms of material efficiency. Since only the material required for the final product is used in the production process, material waste is minimized compared to machining (Gibson et al., 2015). Moreover, thanks to the digital production infrastructure, personalized production and rapid prototyping processes can be carried out effectively.

Another important advantage of additive manufacturing is the reduction in product development time. Conducting the design, prototyping, and testing processes through a single digital chain significantly reduces time to market (Ngo et al., 2018).

ADDITIVE MANUFACTURING TYPES

Additive manufacturing technologies are classified into different categories based on the energy source used, the type of material, and the production mechanism. According to the ISO/ASTM 52900 standard, additive manufacturing methods are classified under main groups such as material extrusion, powder bed fusion, photopolymerization, material deposition, binder jetting, and sheet lamination (ISO/ASTM 52900, 2021).

The general classification of these methods is presented in Figure 2.



	TİP	KATEGORİ	YÖNTEM	TEKNOLOJİ	MALZEME	GÜÇ KAYNAĞI	ÖZELLİKLER
EKLEMELİ İMALAT	SIVI	Eriyik	Malzeme ekstrüzyonu	FDM	Termoplastik	Termal Enerji	-Düşük yazıcı maliyeti -Çoklu malzeme ile baskı -Yüksek mukavemet -Düşük parça çözünürlüğü -Zayıf yüzey iş-sonu -Düşük baskı hızı
		Polimerize edilebilir	Fotopolimerizasyon	SL (SLA)	Fotopolimer, Seramik	UV ışını	-Yüksek baskı hızı -Yüksek parça çözünürlüğü -Yüksek detay -Malzeme maliyeti yüksek
				DLP		Projeksiyon	-Yüksek baskı hızı -Çözünürlük,projeksiyonun piksel boyutuyla sınırlıdır
			Malzeme püskürtme	PJ	Fotopolimer, Wax	UV ışını	-Çoklu malzeme ile baskı -İyi yüzey iş-sonu -Yüksek doğruluk -Yüksek detay
	KATI	Yapışık objeler	Sac laminasyon	LOM	Kağıt Plastik film, Metalik sac, Seramik bant	Lazer Işını	-İyi yüzey iş-sonu -Yazıcı, malzeme, proses maliyeti düşük -Büyük boyutlu malzeme basabilme -Dikey yönde zayıf mukavemet
	TOZ	Eritme	Toz yataklı eritme	SLS	Poliamid, Polimer	Yüksek-Güçlü Lazer Işını	-Yüksek doğruluk -Yüksek detay -Tam dolu parça üretimi -Yüksek mukavemet -Destek yapıları gerekmez
				DMSL	Metal tozu, Seramik tozu		
				SLM			
				EBM		Elektron Işını	
			Direk enerji depolama	LENS	Erimiş metal tozu	Lazer Işını	-Hasarlı ve aşınmış parçaları tamir edebilme -Son-işlem gerekir
				EBAM			
		Yapıştırma	Yapıştırıcı püskürtme	BJ	Seramik tozu, Metal tozu, Kum	Termal Enerji	-Renkli obje baskısı -Destek yapıları gerekmez -Geniş malzeme seçeneği -Yüksek baskı hızı -Son-işlem için infiltran malzeme gerektirir -Düşük dayanım -Yüksek gözeneklilik

Figure 2: Additive Manufacturing Methods (Sürmen, 2019)

Each method has its own advantages and limitations in terms of surface quality, mechanical properties, material diversity, and production cost (Gibson et al., 2015).

In this chapter, FDM technology, which is frequently preferred in academic and industrial applications commonly used in thermoplastic-based production, is discussed in detail in the following chapter.

FDM (FUSED DEPOSITION MODELING)

FDM is an additive manufacturing method based on extruding molten thermoplastic filament from a nozzle at a specific temperature and stacking layers on top of each other. It is widely preferred in academic and industrial applications due to its low-cost equipment, wide range of material options, and ease of use.

Figure 3: Fortus 900 mc FDM Device

HOW DOES FDM PRODUCTION WORK?

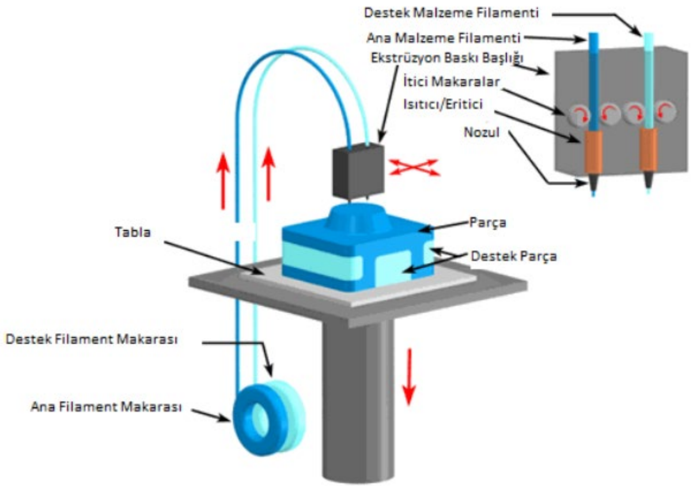


Figure 4: FDM Components (Anonymous, 2008)

First, the 3D CAD file drawn with engineering programs is saved in .STL format. Thanks to the software that manages the entire pre-print digital preparation process for parts to be produced on FDM-type 3D printers, the CAD data is separated into layers, print paths (tool paths) are created, and support structures and production parameters are controlled in detail. The production layout format is determined and transferred to the 3D printer. After the relevant data is opened on the 3D printer, production parameters such as material, color, and layer thickness are determined. If a support geometry is required for the production of the desired part, this support geometry is determined; if not, the main geometry is used.

In the FDM method, production quality is directly dependent on the accuracy of the pre-print preparation stages. The geometric properties determined during the design stage, STL file integrity, slicing parameters, and material conditions significantly affect the repeatability and mechanical performance of the printing process.

Parameters Used in FDM Devices

In the Fused Deposition Modeling (FDM) production process, the mechanical properties, surface quality, and dimensional accuracy of the final product are directly related to the process parameters determined during production. These parameters determine the functional properties of the product by creating the interlayer bonding structure and the microstructure of the part. (Turner & Gold, 2015; Ngo et al., 2018).

Nozzle Temperature

Nozzle temperature is one of the most critical parameters determining the degree of melting of the thermoplastic filament and the extrusion quality. Insufficient nozzle temperature causes the filament to not melt completely and weak interlayer adhesion, while excessively high temperatures can lead to material degradation, flow problems, and surface defects. Optimal nozzle temperature improves mechanical strength by increasing interlayer diffusion (Gibson, Rosen & Stucker, 2015; Turner & Gold, 2015).

Bed Temperature

Bed temperature controls the adhesion of the first layers of the print to the bed surface and the warping effect that may occur throughout the part. Low bed temperatures weaken the adhesion of the first layer and cause lifting at the edges of the part, while an appropriate bed temperature increases dimensional stability by reducing thermal stresses (Ngo et al., 2018). This effect is particularly noticeable in ABS and high-performance thermoplastics.

Layer Thickness

Layer thickness directly affects the surface roughness and production time of parts produced using the FDM method. Thin layer thicknesses provide smoother surface quality and better geometric accuracy, but extend production time. Thicker layers shorten production time; however, they can reduce the interlayer bonding area, leading to a decrease in mechanical properties (Gibson et al., 2015).

Infill Density

Infill density indicates how full the internal structure of the part is and plays a decisive role in the part's weight, mechanical strength, and energy absorption capacity. Low infill densities allow for the production of lightweight and economical parts, while high infill densities increase compressive and bending strength in particular. However, the infill geometry and pattern also significantly affect mechanical performance (Ngo et al., 2018; Torrado Perez et al., 2014).
You can see different pattern structures in Figure 5.

Figure 5: General Overview of the Sample Study Produced in Different Patterns

Print Speed

Print speed determines the time it takes for the extruded material to spread across the layers and has a direct impact on production quality. While high print speeds reduce production time, they can prevent sufficient thermal bonding between layers and cause surface quality to deteriorate. Lower print speeds, on the other hand, improve mechanical properties by enabling more homogeneous layer formation (Turner & Gold, 2015).

Orientation Angle (Print Direction)

The orientation angle refers to how the part is positioned relative to the print bed and causes anisotropic behavior in the mechanical properties of FDM parts. Whether the load-bearing direction is parallel or perpendicular to the layers has a decisive effect on tensile and flexural strength. Parts loaded in a direction parallel to the layers generally exhibit higher strength, while parts loaded in a direction perpendicular to the layers have an increased risk of fracture (Ahn et al., 2002; Domingo-Espin et al., 2015).

While each of these parameters is important on its own, their interaction also plays a critical role in determining the final part quality in the FDM production process. Therefore, it is necessary to determine the optimal combination of parameters for the targeted application.

MATERIALS USED IN FDM

The materials used in Fused Deposition Modeling (FDM) technology are typically thermoplastic polymers supplied in filament form. These materials must be capable of melting within a specific temperature range to be extruded through the nozzle and maintaining their shape stability after cooling. The thermal, mechanical, and rheological properties of materials used in FDM directly affect the stability of the production process and the performance of the final part (Gibson, Rosen & Stucker, 2015).

FDM materials are generally classified into three main groups: standard thermoplastics, engineering plastics, and high-performance thermoplastics.

Standard Thermoplastics

Standard thermoplastics are among the first widely used materials in FDM technology due to their low printing temperatures, easy workability, and low cost advantages. The most commonly used materials in this group are PLA (Polylactic Acid) and ABS (Acrylonitrile Butadiene Styrene).

PLA is often preferred for prototyping and visual models due to its biodegradable structure, low melting temperature, and low deformation tendency during printing. However, PLA's low heat resistance and brittle structure limit its use in engineering applications (Tymrak, Kreiger & Pearce, 2014).

ABS offers higher toughness and impact resistance, but it is a more challenging material in terms of process control due to its requirement for high bed temperatures during printing and its tendency to warp. Despite this, it is widely used for functional prototypes and mechanical test specimens (Ahn et al., 2002).

Engineering Thermoplastics

Engineering thermoplastics offer higher mechanical strength, wear resistance, and chemical stability compared to standard polymers. Materials such as PETG, Nylon (PA6, PA12), and TPU stand out in this group.

PETG offers higher impact resistance and chemical resistance compared to PLA, while exhibiting lower warping tendency than ABS. These properties make it preferred for functional parts and semi-structural applications (Ngo et al., 2018).

Nylon-based filaments, on the other hand, are mechanically advantageous due to their high toughness, wear resistance, and fatigue resistance. However, due to their hygroscopic nature, they require drying before printing and are sensitive to process parameters (Turner & Gold, 2015).

High-Performance Thermoplastics

High-performance thermoplastics play a significant role in the integration of FDM technology into industrial and critical applications. This group includes polymers such as PEEK, PEKK, and Ultem (PEI), which possess high-temperature and high-strength properties.

Ultem (Polyetherimide, PEI) is widely used in aerospace and defense industry applications due to its high glass transition temperature, flame-retardant properties, and superior mechanical performance. Ultem parts produced using the FDM method offer significant weight advantages over their metal counterparts while demonstrating resistance to harsh environmental conditions (Gibson et al., 2015; Ngo et al., 2018).

The production of these materials using FDM requires high nozzle and build plate temperatures and a controlled printing environment, necessitating the precise optimization of process parameters. Otherwise, quality issues such as weak interlayer bonding and internal stress formation may arise.

Filled and Composite Filaments

In FDM technology, fiber or particle-filled composite filaments are also used to enhance mechanical and functional properties. These filaments, containing carbon fiber, glass fiber, or mineral fillers, provide high rigidity and dimensional stability but may cause increased anisotropic behavior (Torrado Perez et al., 2014).

The use of composite filaments enables the integration of FDM parts into structural applications; however, it also brings additional challenges such as nozzle wear and process complexity.

In this context, the selection of materials to be used in FDM should be made considering the targeted mechanical performance, thermal resistance, environmental conditions, and production costs.

DISCUSSION

The findings and literature reviews discussed in this chapter clearly demonstrate that the position of additive manufacturing technologies within modern manufacturing systems should be evaluated as a complementary and supportive production approach rather than a direct alternative to traditional manufacturing methods. The process parameters and material selection criteria examined specifically for the Fused Deposition Modeling (FDM) method show that the final product performance is influenced not only by the production technology used but also by the holistic consideration of the design–material–process interaction (Gibson, Rosen & Stucker, 2015). This situation reveals that FDM technology should be evaluated with a system-oriented approach rather than a “device-oriented” one.

The design freedom offered by additive manufacturing, the ability to produce complex geometries, and the opportunity for rapid prototyping thanks to the digital production infrastructure provide significant advantages over traditional manufacturing methods. However, as emphasized in the literature, surface quality, production speed, and, in particular, the anisotropic mechanical behavior associated with layered structures limit the suitability of additive manufacturing for some critical applications on its own (Ngo et al., 2018). In this context, the industrial use of additive manufacturing is often considered in conjunction with hybrid production approaches; parts produced using FDM are subsequently supported by machining or surface finishing processes. This trend demonstrates that the current limitations of FDM technology are managed through appropriate engineering solutions rather than being completely eliminated.

It has been observed that the mechanical and thermal properties of parts produced using the FDM method are highly dependent on process parameters such as nozzle temperature, layer thickness, print orientation, and infill density. Studies in the literature reveal that the printing direction and interlayer bonding quality, in particular, play a decisive role in tensile and flexural strength (Ahn et al., 2002; Turner & Gold, 2015). This indicates that parts produced by FDM should not be evaluated as isotropic materials and that the relationship between load directions and print direction must be considered during the design phase. From this perspective, not only parameter optimization but also application-specific design strategies are critical for improving the mechanical performance of FDM parts.

From a material perspective, FDM technology was initially limited to standard thermoplastics; however, today, with the integration of engineering and high-performance thermoplastics into the system, its application areas have significantly expanded. In particular, the use of polymers with high temperature resistance, flame retardant properties, and dimensional stability, such as Ultem (PEI), enables the evaluation of FDM technology in sectors requiring high reliability, such as defense and aerospace (Gibson et al., 2015). However, the high nozzle and bed temperatures, controlled printing environments, and narrow process windows required during the processing of such materials increase the complexity of the production process and make process stability a critical parameter.

In this context, for FDM technology to be used effectively and reliably in industrial applications, it is not sufficient to simply select the appropriate material; it is also necessary to systematically optimize process parameters and adopt application-specific design approaches. Existing studies in the literature show that parameter-property relationships have been defined for specific materials and limited experimental conditions; however, these relationships have not yet been established within a generalizable framework for all material groups and loading conditions (Ngo et al., 2018; Torrado Perez et al., 2014). This situation demonstrates that FDM technology remains a dynamic field open to research and development.

In conclusion, the findings discussed in this section show that additive manufacturing, and FDM technology in particular, is a multidisciplinary production approach that should be evaluated alongside traditional manufacturing methods. In the future, the integration of advanced material systems, real-time process monitoring techniques, and numerical modeling methods into the production process will enable FDM-based manufacturing to be adapted to more reliable, repeatable, and high-performance applications. These developments are expected to enable FDM technology to move beyond the prototyping stage and be used more widely in functional and structural applications.

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CHAPTER 2

DEVELOPMENT OF EXTREME TEMPERATURE RESISTANT CERAMIC SYSTEMS AND MODERN PROCESSING APPROACHES

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INTRODUCTION

Ceramic materials capable of withstanding extreme temperatures constitute one of the most strategic areas of modern engineering. These materials have a broad range of applications, from aerospace engineering to nuclear energy systems, from high-speed transportation to protective coatings exposed to thermo-mechanical loading. Key properties such as hardness, high melting temperature, oxidation resistance, and chemical stability sharply distinguish these materials from conventional metals and polymer-based systems. Therefore, the design, fabrication, and performance investigation of materials selected for operation under extreme-temperature conditions represent one of the priority directions of contemporary scientific research. The development of such materials is not limited only to producing components with high melting points. For effective industrial implementation, controlling the microstructure, reducing porosity during sintering, maintaining phase transformations under control, and ensuring stability of mechanical properties are of great importance. In particular, powder-based manufacturing technologies—pressing, sintering, reactive synthesis, SPS, and other modern sintering methods—have enabled major advances in this field. The behavior of extreme-temperature materials is also shaped directly by the scientific principles governing these processing routes. Recent studies indicate that, for ceramic systems operating under extreme conditions, high-temperature resistance alone is not sufficient; the relationship between microstructure and mechanical behavior is also critically important both theoretically and practically. Parameters such as grain size, phase distribution, hardness-to-modulus ratio, thermal expansion, and resistance to cracking are among the main factors that determine overall performance. The aim of research in this direction is to form ceramic systems that preserve both mechanical and chemical stability at high temperatures. This review aims to analyze the development of extreme-temperature-resistant ceramic materials within a broad scientific context, while also examining their processing technologies, microstructural evolution, and performance indicators. Here, comparisons of various sintering methods, application areas of refractory ceramic systems, their advantages and limitations, as well as the influence of modern scientific approaches on future development directions are evaluated. Since such materials play a decisive role in meeting the high demands of modern industry, analyzing them on a scientific basis is of great importance both academically and practically.

Scientific Fundamentals and Key Advantages of Extreme-Temperature Ceramics

Extreme-temperature-resistant ceramic materials occupy a special place in materials science and are characterized as material systems that maintain stability under high heat, mechanical loading, and aggressive chemical environments. These materials are generally classified as refractory and ultra-high temperature ceramics and possess properties that exceed the temperature limits of conventional metal-based materials (Kingery, Bowen & Uhlmann, 1976). The continuous increase in temperature requirements in modern industry and technology further enhances the scientific and practical significance of these ceramics. Demand for such materials is rapidly growing, especially in aerospace, energy, and high-temperature process industries. One of the main distinguishing features of extreme-temperature ceramics is their high melting temperatures and strong covalent or ionic–covalent bonding structures. The strength of interatomic bonding ensures that the crystal lattice remains stable at high temperatures and prevents structural degradation under prolonged thermal exposure. These bonding mechanisms limit atomic mobility, slow down diffusion processes, and enable functional properties to be preserved at high temperatures. As a result, such ceramic materials serve as reliable structural bases for components operating under extreme conditions. These materials can retain a significant portion of their mechanical properties even at elevated temperatures and demonstrate advantages in hardness, wear resistance, and resistance to frictional effects. While many materials exhibit rapid mechanical weakening as temperature rises, extreme-temperature ceramics show greater resistance to such effects. Particularly in applications involving aero-thermal loading and high-speed environments, these characteristics are considered among the primary criteria for material selection and directly affect long-term service performance of components. In terms of thermal behavior, extreme-temperature ceramics are typically characterized by low coefficients of thermal expansion and high thermal stability. These features reduce internal stresses generated under rapidly changing temperature conditions and improve resistance to thermal shock. Moreover, stable heat transfer at high temperatures supports preservation of structural integrity and helps prevent microstructural damage (Kingery, 1955). These properties play a decisive role in the reliability of ceramic components operating in extreme environments.

The performance of extreme-temperature ceramics is not limited only to chemical composition; it is also closely related to the microstructural characteristics of the material. Crystallite size, phase distribution, and porosity level directly affect mechanical strength, thermal stability, and long-term service behavior. A denser and more homogeneous microstructure enables more uniform stress distribution and reduces the probability of crack formation. Therefore, controlled microstructure formation and optimization during processing are considered among the key scientific approaches for developing high-performance ceramic systems (German, 1996). Overall, extreme-temperature ceramics offer unique advantages for applications where high temperature, mechanical loading, and aggressive environments coexist. A deep understanding of their scientific foundations and proper establishment of structure–property relationships provide a solid scientific basis for the design and implementation of future high-performance ceramic systems.

Thermal and Chemical Behavior of Ceramic Materials

One of the main factors that determines the application potential of extreme-temperature-resistant ceramic materials is their behavior in high-temperature and aggressive chemical environments. High heat flux, oxidizing atmospheres, and rapid temperature variations can significantly affect structural stability and service life. Under such conditions, not only the mechanical strength but also the thermal and chemical stability of the material plays a decisive role. Therefore, analyzing the thermal and chemical properties of ceramic materials in a general framework is considered one of the key stages in material selection for extreme environments. Ceramic materials are typically characterized as systems that preserve structural stability under high-temperature conditions. This property is associated with strong interatomic bonding within the crystal lattice and results in limited atomic diffusion at high temperatures. Reduced atomic mobility allows the microstructure to remain stable under prolonged thermal exposure. Consequently, ceramic materials can retain a substantial portion of their mechanical and physical properties even during long-term high-temperature operation. In addition, a low coefficient of thermal expansion reduces internal stresses generated under rapidly changing temperature conditions and decreases the risk of crack formation due to thermal shock (Kingery, Bowen & Uhlmann, 1976; Kingery, 1955). In extreme-temperature applications, resistance to thermal shock is particularly important for reliability. During rapid heating-cooling cycles, temperature gradients inside the material generate internal stresses. If these stresses cannot be effectively accommodated by the material, cracks may form and structural degradation may occur. In ceramic materials, microstructural homogeneity and low thermal expansion play an important role in preventing such damage. Beyond thermal stability, the ability of ceramic materials to dissipate or redistribute heat under extreme conditions also influences their long-term performance. Non-uniform heat distribution may result in localized overheating, which can accelerate microstructural damage and initiate premature failure. Therefore, thermal conductivity and heat transport mechanisms within the ceramic matrix should be considered alongside thermal expansion behavior. Materials exhibiting balanced thermal conductivity can reduce temperature gradients and mitigate thermally induced stresses, thereby enhancing resistance to both thermal shock and long-term degradation in extreme environments.

The chemical behavior of ceramic materials in high-temperature applications-especially oxidation processes-also plays a critical role. In oxidizing environments, surface layers formed on the material may either increase durability by acting as protective barriers or accelerate structural degradation. Formation of protective oxide layers can limit contact between the aggressive environment and internal regions, thereby extending service life. Conversely, unstable and porous oxide layers may lead to deterioration of mechanical and thermal properties. In this regard, proper selection of chemical composition and microstructure enables reduction of oxidation rates and ensures long-term stability.

Chemical degradation at elevated temperatures is often accompanied by coupled thermo-chemical effects, where oxidation and diffusion processes interact with microstructural features such as grain boundaries and pores. These coupled mechanisms may result in progressive weakening of the material, even if the initial oxidation rate is relatively low. Therefore, long-term chemical stability should be evaluated not only through short-term oxidation resistance, but also through the material's ability to maintain structural integrity under sustained exposure to reactive environments. Recent studies indicate that nanostructured ceramic materials may exhibit superior thermal and chemical behavior compared to conventional ceramic systems under certain conditions. In systems with nanoscale crystallites, more homogeneous microstructure formation and changes in defect distribution mechanisms can delay crack initiation and improve overall durability. Furthermore, changes in diffusion behavior within nanostructured systems may also influence chemical degradation mechanisms occurring at high temperatures. However, maintaining these advantages depends on preserving the nanostructure at elevated temperatures, which makes appropriate selection of processing technologies essential (Gleiter, 2000).

Processing Technologies and Sintering Mechanisms

The functional properties of extreme-temperature-resistant ceramic materials are closely related not only to their chemical composition but also to the applied processing technologies. The methods selected during manufacturing directly influence density, microstructure, and behavior at high temperatures. Therefore, proper selection of processing technologies is considered a decisive stage in producing high-performance ceramic systems. In conventional processing approaches, sintering ceramic powders at high temperatures is used as the primary mechanism. During this process, diffusion leads to bonding between particles and increases material density. Although classical sintering methods are widely used, their requirement for very high temperatures may cause grain growth and non-uniform microstructure formation. This can limit the mechanical and thermal stability required for extreme-temperature applications. The development of modern processing technologies aims to overcome these limitations. Rapid sintering approaches enable achieving high density in shorter times and allow more effective control of microstructure. These methods help reduce porosity, increase mechanical strength, and obtain more stable behavior at high temperatures. As a result, the reliability of ceramic components operating under extreme-temperature conditions increases significantly.

Production of nanoceramic materials requires more sensitive process control compared to conventional ceramic systems. Although materials with nanoscale crystallites can exhibit high mechanical strength and improved thermal stability, rapid crystallite growth at high temperatures may eliminate these advantages. Therefore, preserving the nanostructure is considered one of the main scientific and technological challenges at the processing stage. As reported in the literature, nanostructured ceramic systems can show superior performance relative to conventional ceramics under certain conditions (German, 1996; Gleiter, 2000). The choice of processing technologies also directly affects the scalability of ceramic materials and the expansion of their application fields. When transitioning ceramic systems that exhibit high performance at laboratory scale to industrial scale, factors such as process repeatability, energy efficiency, and cost optimization become particularly important. In this regard, modern sintering and densification technologies not only improve material properties but also enable large-scale and reliable production of ceramic components intended for extreme-temperature applications (Rahaman, 2003).

Microstructure–Performance Relationship

The mechanical, thermal, and chemical behavior exhibited by ceramic materials under extreme-temperature conditions is directly related to their microstructural characteristics. The concept of microstructure encompasses parameters such as crystallite size, phase distribution, porosity level, the nature of grain boundaries, and the type and distribution of existing defects within the material. These microstructural parameters are considered the primary factors that determine both the short-term mechanical response and the long-term thermal stability of ceramic materials. For ceramic components operating in extreme environments, the role of microstructure becomes even more critical, as material failure under such conditions often originates from crack initiation and propagation at the microstructural level. Controlling crystallite size and phase distribution plays a crucial role in the development of mechanical strength and crack resistance in ceramic materials. A more homogeneous microstructure promotes a more uniform distribution of internal stresses within the material and delays the onset of crack initiation. In addition, reduction of porosity increases material density, enhances load-bearing capability, and enables more stable heat transfer behavior. As reported in the literature, purposeful optimization of microstructure can significantly improve the reliability and service life of ceramic systems designed for extreme-temperature applications (German, 1996). In particular, better control over pore size and distribution reduces the likelihood of mechanical weakening and thermal damage. The behavior of ceramic materials under extreme-temperature conditions is not limited to mechanical strength alone; it is also governed by diffusion processes occurring at elevated temperatures and resistance to thermal shock. Phase boundaries and crystallite size within the microstructure directly influence the kinetics of these processes. A finer and more uniformly distributed microstructure can reduce localized stresses generated by temperature gradients and help prevent structural degradation. In this context, the response of the microstructure to repeated heating–cooling cycles is of particular importance. Recurrent thermal cycling may lead to the accumulation of residual stresses and accelerate the formation of microcracks. Therefore, maintaining microstructural stability under thermal cycling is considered one of the key requirements for materials intended for extreme-temperature applications Grain boundaries may play both beneficial and limiting roles in the behavior of ceramic materials. On one hand, grain boundaries can alter crack propagation paths, absorb fracture energy, and increase resistance to damage by deflecting or blunting cracks. On the other hand, at high temperatures, diffusion and chemical reactions often intensify along grain boundaries, making their characteristics critical for long-term stability. Consequently, the number, nature, and distribution of grain boundaries and interphase interfaces must be carefully considered during microstructural design, as they strongly influence overall material performance. In addition, the influence of microstructure on chemical stability should be taken into account. At elevated temperatures, oxidation and other chemical reactions occur primarily at the material surface and along grain boundaries; therefore, the microstructural characteristics directly affect the rate and mechanism of these processes. A denser and more homogeneous microstructure can limit the contact area between the material and oxidizing environments, thereby slowing chemical degradation. Conversely, high porosity facilitates penetration of reactive species into the material, accelerating chemical degradation and leading to earlier loss of mechanical and thermal properties. This aspect is particularly important for ensuring the long-term stability of ceramic components operating in extreme environments.

In nanostructured ceramic materials, the microstructure–performance relationship becomes more complex and multidimensional. Nanoscale crystallites can alter defect propagation mechanisms, thereby increasing resistance to cracking, thermal damage, and mechanical loading. At the same time, enhanced microstructural homogeneity in nanostructured systems may enable more effective retention of mechanical and thermal properties at high temperatures. However, the strong tendency for crystallite growth at elevated temperatures threatens the stability of the nanostructure. As a result, preservation of microstructure in such materials is closely linked to the applied processing technologies (Gleiter, 2000). From this perspective, the main objective in the practical application of nanostructured ceramic systems is not only the initial formation of a nanostructure, but also its stabilization under prolonged high-temperature exposure. Overall, improving the performance of ceramic materials requires purposeful microstructural design. Optimization of crystallite size, porosity, and phase composition represents one of the main strategies for enhancing resistance to mechanical loading, thermal shock, and chemical degradation. In this regard, in-depth investigation of the microstructure–performance relationship has fundamental importance for the development of more durable and high-performance ceramic systems intended for extreme-temperature applications. Future research efforts are expected to focus on more precise control of microstructural parameters and a deeper understanding of how these parameters evolve under different extreme service conditions.

Material Requirements for Extreme-Temperature Applications

Selecting materials for extreme-temperature applications is considered one of the most complex and multi-criteria problems in modern materials science and engineering. In such applications, materials are exposed not only to high temperature, but also to mechanical loading, rapid temperature changes, oxidizing and aggressive chemical environments, and in some cases radiation. Therefore, material selection for extreme environments should be based not on a single property, but on satisfying a set of interrelated thermal, mechanical, chemical, and structural requirements in an integrated manner. One of the main requirements for materials used at high temperatures is thermal stability. The material must maintain structural integrity under thermal exposure and should not exhibit phase transformations or rapid degradation processes. In addition, properties such as a low coefficient of thermal expansion and high thermal conductivity reduce internal stresses generated by temperature gradients and improve resistance to thermal shock. For components operating in extreme-temperature applications, these features are among the main factors determining service life and reliability (Kingery, Bowen & Uhlmann, 1976). Mechanical properties are also an integral part of material requirements for extreme-temperature applications. At high temperatures, mechanical strength, hardness, and resistance to creep should be preserved. While many materials have satisfactory mechanical properties at room temperature, these properties may deteriorate rapidly as temperature increases. Therefore, materials intended for extreme environments must be able to carry mechanical loads at high temperatures and must not undergo structural weakening during long-term service. Chemical durability is particularly important in extreme-temperature environments. At high temperatures, oxidation, evaporation, and other chemical reactions may lead to formation of surface layers that are either protective or cause degradation. The chemical composition and microstructural characteristics directly affect the rate and nature of these processes. Materials that behave stably in oxidizing environments are considered more advantageous for extreme-temperature applications, because chemical degradation can cause loss of mechanical and thermal properties (Fahrenholtz & Hilmas, 2007). From a structural perspective, microstructural characteristics are among the key requirements for extreme-temperature applications. Crystallite size, phase distribution, and porosity level directly affect both thermal and mechanical behavior. A denser and more homogeneous microstructure enables more uniform stress distribution and reduces the probability of crack formation. These features are particularly important for applications involving rapid heating–cooling cycles. Nanostructured material approaches provide new opportunities for meeting material requirements in extreme-temperature applications. In materials with nanocrystalline structures, changes in defect propagation mechanisms and more homogeneous microstructure formation may enable superior mechanical and thermal behavior under certain conditions. However, preserving nanostructure at high temperatures remains one of the main challenges for practical application and requires optimization of processing technologies in line with these requirements (Gleiter, 2000). Material requirements for extreme-temperature applications are not limited to technical properties; they should also be evaluated in terms of manufacturing and implementation. Industrial scalability, process repeatability, and economic efficiency must be considered. Materials demonstrating high performance at laboratory scale may face additional technological challenges when transferred to industrial scale, and this factor plays an important role in material selection. Overall, material requirements for extreme-temperature applications comprise multi-dimensional and closely interconnected criteria.

Thermal stability, mechanical strength, chemical durability, and structural integrity form the core of these requirements. Meeting these criteria in an integrated manner has fundamental importance for developing reliable and long-lasting material systems under high-temperature conditions, and it necessitates more detailed study of specific material systems, including carbide-based ceramics.

Example of Ceramic Systems for Extreme-Temperature Applications

Selecting structural and functional materials operating under extreme-temperature conditions is one of the most challenging problems in modern materials science and engineering. In such environments, materials are simultaneously exposed to high heat flux, mechanical loading, oxidizing and aggressive chemical atmospheres, and in some cases radiation. Because conventional metal-based materials often exhibit mechanical weakening, rapid oxidation, and structural degradation under these conditions, interest in ceramic-based material systems for extreme-temperature applications has increased significantly. Carbide-based ceramic materials occupy a special position among ceramic systems intended for extreme-temperature applications. These materials are distinguished by high melting temperature, high hardness, wear resistance, and structural stability at elevated temperatures. The strong covalent bonding structure of carbides enables them to maintain mechanical and thermal properties relatively stable at high temperatures. These characteristics make carbide-based ceramic systems promising for aero-thermal protective coatings, nose cones of high-speed vehicles, thermal shields, and other engineering components exposed to extreme heat flux. Zirconium-based carbide ceramic systems are particularly notable in terms of high-temperature resistance and structural stability. Their high melting temperature and low diffusion rates are among the main factors preventing structural breakdown under extreme thermal conditions. Moreover, the behavior of such ceramic systems depends not only on chemical composition but also closely on microstructural properties. Crystallite size, porosity level, and phase homogeneity are considered key factors shaping mechanical and thermal behavior at high temperatures. Recent studies suggest that carbide-based ceramic systems with nanocrystalline structures may exhibit different—and in some cases superior-behavior compared with conventional micro-scale structures. In materials with nanoscale crystallites, defect propagation mechanisms and diffusion processes change, which can delay crack initiation and propagation. This feature is particularly important for materials operating in extreme-temperature and radiation environments and makes nanocrystalline ceramic systems an active research subject. To study the behavior of zirconium-based nanocrystalline carbide particles under high temperature and radiation, various experimental and analytical approaches are applied. In such studies, methods such as infrared spectroscopy, differential scanning calorimetry, and thermogravimetric analysis are used to evaluate the general nature of thermal processes and structural changes. These methods enable deeper understanding of behavior mechanisms at high temperatures and provide an important scientific basis for material design for extreme environments (Huseynov et al., 2023). The literature includes numerous studies describing carbide-based ceramic systems as promising materials for extreme-temperature applications. These studies commonly discuss high-temperature stability, oxidation behavior, and the role of microstructure. In particular, it is emphasized that carbide systems belonging to the class of ultra-high temperature ceramics (UHTCs) have significant potential for aerospace applications. In this regard, zirconium-based carbide ceramic systems are considered one of the major research directions among UHTC materials (Fahrenholtz & Hilmas, 2007). Overall, zirconium-based carbide ceramic systems are widely investigated as candidate materials for use under extreme-temperature and radiation conditions. Research in this area aims to more precisely identify the relationships between microstructural properties and thermal/chemical behavior. The formation and preservation of nanocrystalline structures is regarded as one of the key scientific and technological directions that can expand the use of these materials in future high-temperature applications.

Conclusions and Future Perspectives

In this review, the scientific foundations, structural characteristics, and performance indicators of ceramic materials applied under extreme-temperature conditions have been evaluated within the framework of modern materials science. The literature overview indicates that, in environments where high temperature, mechanical loading, and aggressive chemical conditions coexist, conventional metal-based materials often lose their functional properties. Therefore, ceramic-based material systems become more suitable alternatives for extreme environments. In particular, refractory and carbide-based ceramic materials attract broad research interest in terms of high-temperature stability and structural durability. The influence of processing technologies on the final performance of ceramic materials has been highlighted as one of the main topics of this review. Proper selection of sintering mechanisms, temperature regimes, and process parameters directly affects density, porosity, and microstructural homogeneity. Controlled microstructure formation plays an important role not only in mechanical strength but also in ensuring thermal stability and long-term service reliability. This approach significantly expands the functional capabilities of ceramic systems intended for extreme-temperature applications. Analysis of the microstructure–performance relationship shows that crystallite size, phase distribution, and porosity level are among the main parameters shaping the behavior of ceramic materials. A denser and more homogeneous microstructure enables more uniform stress distribution and reduces the probability of crack formation. This supports preservation of structural integrity under high temperature and rapid temperature changes. Microstructure-oriented design can therefore be considered a key strategy for reliable use of ceramic components operating in extreme environments (Kingery, Bowen & Uhlmann, 1976). Growing scientific interest in nanostructured ceramic materials has opened new perspectives in this field. In ceramic systems with nanocrystalline structures, changes in defect propagation mechanisms and more homogeneous microstructure formation may enable superior mechanical and thermal behavior under certain conditions. Nevertheless, maintaining nanostructure stability at high temperatures remains one of the main practical challenges. This requires further improvement of processing technologies and development of approaches that ensure long-term microstructural stability. Carbide-based ceramic systems, especially materials within the class of ultra-high temperature ceramics, are of particular importance for extreme-temperature applications. Their behavior under high temperature, oxidation, and mechanical loading determines their potential for aerospace, energy, and other advanced technological fields. Studies indicate that the performance of such ceramic systems is strongly linked to microstructural properties, and deeper investigation of these relationships provides an important scientific basis for future material design (Gleiter, 2000; Fahrenholtz & Hilmas, 2007).

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CHAPTER 3

CONTEMPORARY SCIENTIFIC CONCEPTS FOR ENHANCING FUNCTIONAL PERFORMANCE IN HETEROSTRUCTURES

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INTRODUCTION

In recent decades, the rapid scientific and technological progress in the field of nanomaterials and low-dimensional systems has fundamentally transformed classical approaches in materials science. The discovery of two-dimensional (2D) materials composed of atomically ordered layers, along with the ability to combine these materials in various configurations, has positioned the concept of heterostructures at the forefront of modern materials research (Geim & Grigorieva, 2013; Novoselov et al., 2016) . In such systems, functional properties are governed not only by chemical composition but also by interlayer interactions, interface engineering, structural modifications, and external stimuli.

Van der Waals (VdW) heterostructures enable the integration of materials with dissimilar lattice constants within a single structure due to weak interlayer bonding. This approach allows for the purposeful tuning of electronic, optical, mechanical, and thermal properties of materials (Geim & Grigorieva, 2013). Consequently, heterostructure engineering represents a paradigm shift in materials science—from the concept of “property design” toward the scientifically guided control of material properties.

This study provides a comprehensive and systematic overview of contemporary scientific concepts employed to enhance functional performance in heterostructures. Particular emphasis is placed on intercalation methodologies, copper (Cu) intercalation in GaSe-based heterostructures, the effects of irradiation on structural and functional properties, as well as the scientific and industrial significance of the author’s original research.

Crystallographic Fundamentals and Physical Properties of Heterostructures

Two-dimensional (2D) van der Waals (VdW) heterostructures constitute a distinct class of materials with tunable optoelectronic properties and offer broad prospects for the development of next-generation functional devices. These structures are characterized by strong intralayer covalent bonding and weak interlayer van der Waals interactions. This anisotropic bonding nature forms the fundamental physical basis of heterostructure engineering, enabling the integration of materials with different crystallographic structures within a single system.

Compared to mechanical stacking approaches (top-down methods), the direct fabrication of VdW heterostructures via epitaxial growth techniques allows for the formation of cleaner and atomically well-controlled interlayer interfaces. In this context, van der Waals epitaxy plays a particularly important role (Geim & Grigorieva, 2013). During the synthesis of two-dimensional GaSe atomic layers on graphene (Gr) substrates via van der Waals epitaxy, it has been observed that the crystal orientation is not random but is strongly correlated with the morphological features of the substrate. Specifically, the orientation of wrinkles present on the graphene surface directs the growth orientation of GaSe nuclei, resulting in the formation of preferential crystallographic alignments.

In the initial stage of the VdW epitaxial growth process, numerous GaSe nuclei are formed; however, nuclei possessing energetically favorable orientations selectively grow, coalesce, and eventually form large-area single-crystalline layers with lateral dimensions reaching several tens of micrometers. Theoretical calculations of interlayer energetics, combined with atomic-resolution scanning transmission electron microscopy (STEM) and electron diffraction measurements, reveal that a relative rotation angle of approximately 10.9° between the GaSe lattice and graphene corresponds to the energetically most favorable van der Waals heterostructure configuration, exhibiting the highest binding energy and the longest-range crystalline ordering (Li et al., 2015).

Such GaSe/Gr VdW heterostructures are characterized by an enhancement of the Raman E_2^1g phonon mode in monolayer GaSe, indicating modifications in interlayer interactions and phonon–electron coupling. Simultaneously, a pronounced quenching of photoluminescence intensity is observed due to efficient charge transfer mechanisms. This behavior confirms the occurrence of effective electron transfer between the GaSe and graphene layers, accompanied by a redistribution of energy levels at the interface.

It should be noted that, despite the significant lattice mismatch between GaSe and graphene, van der Waals epitaxy effectively overcomes this limitation and enables the formation of high-crystalline-quality structures. This represents a key advantage of VdW heterostructures over conventional covalent epitaxial systems. As a result, precise orientation control and the realization of large-area single-crystalline flakes open promising avenues for the scalable synthesis of van der Waals heterostructures, which is essential for the development of advanced optical and optoelectronic devices.

The scientific and technological significance of two-dimensional heterostructures is not limited to lattice-matched material systems. The fabrication of heterostructures from layered semiconductors with substantial lattice mismatch remains a challenging technological task. In this regard, GaSe/$MoSe_2$ heterostructures serve as a model platform for studying misfit systems. The synthesis of such heterostructures via a two-step chemical vapor deposition (CVD) process results in both vertically stacked and laterally aligned structural configurations (Li et al., 2016).

Vertically stacked GaSe/$MoSe_2$ heterostructures exhibit well-defined crystallographic orientation alignment and periodic superlattice structures in accordance with van der Waals epitaxy. In contrast, lateral heterostructures do not display direct epitaxial matching at the material boundary. However, during the CVD process, the advancement of the GaSe monolayer onto the $MoSe_2$ surface leads to the formation of a narrow, vertically stacked van der Waals heterostructure strip along the interface. Consequently, the lateral junction region effectively transforms into a functional interface with vertical heterostructure characteristics.

Experimental investigations demonstrate that vertically stacked GaSe/$MoSe_2$ van der Waals heterostructures facilitate the formation of a p–n junction, accompanied by efficient separation of photogenerated charge carriers across the interface. These structures exhibit gate-tunable photovoltaic responses and possess significant potential for applications in field-effect transistors, high-sensitivity photodetectors, and next-generation solar cells (Li et al., 2016). These findings clearly highlight the strong correlation between the crystallographic foundations of heterostructures and their functional properties.

Intercalation Methodology: Theoretical and Practical Approaches

Intercalation is the process of inserting atoms, ions, or molecular groups into the interlayer space (van der Waals gap) of layered materials (Butler et al., 2013; Coleman et al., 2011). This approach is widely regarded as one of the most versatile and effective methods for the purposeful modulation of functional properties in heterostructures. During intercalation, the interlayer spacing and interaction regime are altered, the electronic charge density is redistributed, and the Fermi level shifts, which can ultimately lead to the emergence of new electronic states and, in some cases, even the formation of new phases.

Physicochemical Foundations: Energy, Charge Transfer, and Interlayer Bonding

The primary "governing parameters" of the intercalation process include the chemical potential of the intercalant (guest phase), the size of the van der Waals gap of the host material, its electronic structure, and defect density. Upon intercalation, weak van der Waals interactions can be partially replaced by stronger, Coulomb-type interactions, which in turn influence mechanical stiffness, the phonon spectrum, and the scattering mechanisms of charge carriers.

In layered materials, intercalation is often accompanied by charge transfer. Donor-type intercalants enhance n-type doping by transferring electrons to the host layers, whereas acceptor-type intercalants can withdraw electrons, thereby strengthening p-type behavior (Butler et al., 2013). As a result of this mechanism:

- electrical conductivity and charge carrier concentration are modified;
- the relative positions of the band edges (band alignment) are reconstructed;
- exciton dynamics and optical transition probabilities are altered;
- local strain and lattice deformation affect the frequencies and linewidths of phonon modes.

Types of Intercalation and Their Functional Outcomes

Intercalation mechanisms are broadly classified into three main categories:

Ionic intercalation

The insertion of ions such as Li^+, Na^+, and K^+ into the interlayer space via electrochemical or chemical routes plays a crucial role in energy storage and conversion technologies. Ionic intercalation can also induce phase transitions in certain two-dimensional semiconductors (e.g., 2H → 1T/1T′), thereby dramatically enhancing the electrical conductivity of the material.

Metallic intercalation

Metallic intercalants such as Cu, Fe, Co, and Ni can give rise to new electronic phases through electron donation, the introduction of localized magnetic moments, or hybridization effects. Metallic intercalation is of particular interest for engineering semiconductor–metal transitions, enabling collective electronic phenomena (including plasmonic response, screening effects, and abrupt shifts in the Fermi level), and tuning optical absorption.

Molecular intercalation

Polar molecules, organic complexes, or solvated ion pairs increase the interlayer spacing and simultaneously modify the dielectric environment within the van der Waals gap. This enables the engineering of optoelectronic properties by influencing exciton binding energies, surface dipoles, and interfacial potential barriers.

This classification is also of practical significance: ionic intercalation predominantly governs energy-related applications and phase transitions; metallic intercalation is central to conductivity modulation and phase engineering; and molecular intercalation plays a dominant role in dielectric and interface engineering.

Kinetics and the Concept of “Staging”: From Heterogeneity to Controlled Systems

Intercalation is not solely a thermodynamic process but also a kinetically governed phenomenon. The diffusion of intercalants depends on the width of the van der Waals gap, the sliding capability of the layers, the reactivity of edges, and the distribution of defects. In layered systems, intercalation often proceeds in a staged manner, wherein intercalants occupy specific layer intervals, giving rise to periodic “intercalation stages.” Because this staging behavior directly influences material properties, it represents one of the critical parameters for achieving controlled intercalation.

As a consequence of heterogeneous intercalation, the local coexistence of distinct phases—intercalated and non-intercalated regions—can occur. Therefore, modern approaches aimed at achieving uniform and homogeneous intercalation place particular emphasis on the following strategies:

- optimization of sample thickness;
- activation of edge sites;
- precise control of the electrochemical potential;
- optimization of temperature and time parameters.

Practical Intercalation Pathways: From Laboratory to Industry

Intercalation methodologies are implemented through several principal practical routes:

Chemical intercalation

In this approach, contact between the intercalant and the host material is established via reactive vapor, melt, or solution phases. Although conceptually simple, this method requires strict control to ensure homogeneity and reproducibility.

Electrochemical intercalation

By controlling the applied potential, the degree of intercalation can be tuned in a stepwise and reversible manner. This technique is particularly advantageous for controlling phase transitions in two-dimensional materials, enabling fine-tuning of carrier concentration and allowing real-time monitoring of the process through in situ measurements.

Electrostatic gating–intercalation hybrids

High-field gating methods (e.g., ionic gating) are in some cases applied simultaneously with or sequentially to intercalation. This represents a modern approach that enables broad-range control of electronic states in heterostructures.

From an industrial perspective, intercalation offers several distinct advantages:

- low-temperature operation, making it more energy-efficient than high-temperature diffusion doping;
- scalability, as it can be integrated with large-area synthesis techniques such as chemical vapor deposition (CVD);
- reversible tunability, enabling cyclic control of material properties through intercalation/deintercalation processes in certain systems;
- preservation of crystalline quality, as properly optimized conditions can avoid severe lattice damage typically associated with bulk doping;
- multifunctional impact, allowing simultaneous modulation of electrical, optical, thermal, and mechanical parameters.

Characterization: Identification and Investigation of Intercalation

Reliable verification of intercalated heterostructures requires a multimodal characterization approach, as no single technique alone can fully capture the structural, chemical, and functional consequences of intercalation. The most commonly employed methods include:

- **X-ray diffraction (XRD):** detection of increased interlayer spacing and identification of staging behavior;
- **Raman spectroscopy:** shifts in phonon modes, changes in linewidths, and sensitive markers of strain and charge transfer;
- **X-ray photoelectron spectroscopy (XPS):** detailed analysis of the chemical state of the intercalant and quantitative evaluation of charge transfer;
- **Scanning transmission electron microscopy (STEM) and electron diffraction:** nanoscale visualization of interlayer structure and localized intercalated phases;
- **Electrical measurements (I–V characteristics, Hall effect, field-effect transistor measurements):** determination of carrier concentration, mobility, and modifications of contact barriers;
- **Optical measurements (photoluminescence, absorption spectroscopy):** probing exciton dynamics, recombination channels, and band alignment.

The purpose of this section is to emphasize that intercalation is not merely an "insertion" process, but rather a systematic methodology for reshaping the energy landscape of layered materials and enabling programmable control over their functional properties.

GaSe-Based Heterostructures and Copper (Cu) Intercalation

As a two-dimensional layered semiconductor, GaSe exhibits high optical sensitivity, strong light–matter interaction, and anisotropic electronic properties. This material is distinguished by its broad spectral light absorption, relatively large exciton binding energy, and layered structure, which makes it highly amenable to functional modification. These characteristics render GaSe a promising platform for heterostructure engineering and various material engineering strategies, including intercalation. In the studies conducted by the author, copper (Cu) intercalation in GaSe-based heterostructures has been systematically and purposefully investigated.

The insertion of Cu atoms into the interlayer space of GaSe directly and multilevelly affects the electronic structure of the system. As a metallic intercalant, copper acts as an electron donor, transferring additional free charge carriers to the GaSe layers and thereby increasing the carrier concentration. This process is accompanied by an upward shift of the Fermi level, modification of the band structure, and, consequently, a significant enhancement of electrical conductivity. In this sense, Cu intercalation serves as an effective tool for gradually driving the semiconducting behavior of GaSe toward a metal-like regime.

The observed increase in conductivity is not solely attributable to the introduction of additional charge carriers. The occupation of the interlayer space by Cu atoms strengthens interlayer coupling, enhances the probability of electron tunneling between layers, and alters carrier scattering mechanisms. As a result, key functional parameters such as carrier mobility and contact resistance are favorably modified. From this perspective, Cu intercalation in GaSe-based heterostructures should be regarded as a comprehensive engineering approach that goes beyond conventional doping by inducing a broader range of functional effects.

Raman spectroscopy measurements clearly reveal frequency shifts and linewidth variations in phonon modes following intercalation. These changes indicate, on the one hand, the strengthening of interlayer interactions and, on the other hand, the formation of local strain fields induced by the incorporation of Cu atoms. Such phonon behavior confirms modifications in electron–phonon coupling and a reconfiguration of energy transfer mechanisms within the structure. Photoluminescence analyses further demonstrate a pronounced quenching of emission intensity as a result of activated charge transfer processes, serving as an important indicator of altered electron–electron and electron–phonon interactions in Cu-intercalated GaSe heterostructures.

One of the key advantages of copper intercalation lies in the technological and economic accessibility of Cu. Compared to rare and costly intercalants, copper is abundant, inexpensive, and already widely utilized in industrial processes. This makes Cu intercalation particularly attractive for transitioning from laboratory-scale studies to real-world applications. Moreover, the chemical stability of copper and its relative robustness in various environments provide an additional advantage for maintaining the long-term operational stability of GaSe-based heterostructures.

A critical survey of the literature indicates that intercalation in GaSe-based heterostructures-particularly copper intercalation—has not yet been extensively explored. While the majority of existing studies focus on transition metal dichalcogenides such as MoS_2 and WS_2, systematic and in-depth investigations of GaSe/Cu intercalation systems remain limited. In this context, the research conducted by the author makes a significant contribution by addressing this fundamental knowledge gap and expanding the functional potential of GaSe-based heterostructures.

In conclusion, Cu intercalation emerges as an effective and promising approach for enhancing electrical conductivity, purposefully tuning the electronic structure, and modifying the optoelectronic response of GaSe-based heterostructures. This strategy not only opens new avenues for fundamental scientific research but also offers realistic and practical opportunities for industry-oriented applications.

Effects of Irradiation on Intercalated Heterostructures

Irradiation serves as an important tool for modifying the structural and functional properties of materials and acts as an “external tuning parameter” in modern materials engineering. In particular, ion, electron, and gamma irradiation are widely employed in heterostructure systems for defect engineering, modulation of charge carrier behavior, and purposeful control of electronic states. In intercalated heterostructures, the effects of irradiation become more complex, as irradiation-induced defects interact with intercalant atoms, giving rise to additional physical mechanisms.

In the studies conducted by the author, a gamma irradiation facility based on the radioactive isotope Co-60 was employed as the irradiation source. The selection of a Co-60 source was motivated by its stable energy spectrum and its ability to generate a homogeneous irradiation field (Abasova et al., 2011). The deep penetration of gamma photons into the material enables uniform volumetric modification of GaSe/Cu intercalated heterostructures, allowing the investigation of fundamental changes that are not limited to surface effects.

The interaction of gamma irradiation with materials is primarily associated with the generation of point defects, such as vacancies, interstitial atoms, and antisite defects, as well as the activation of pre-existing defects. Within low- and intermediate-dose regimes, these defects do not cause severe damage to the crystal lattice; instead, they promote the redistribution of electronic states and the formation of localized energy levels. In intercalated systems, this process acquires a more specific character: irradiation-induced defects can act as additional trapping centers or charge exchange channels for intercalant atoms.

The experimental results demonstrate that the gamma irradiation doses applied in this work correspond to a non-destructive, stimulative dose regime. In other words, the applied doses do not lead to lattice degradation or amorphization; rather, they result in an enhancement of electronic and optical properties. Within this stimulative dose regime, irradiation facilitates:

- an effective increase in charge carrier concentration;
- passivation of recombination centers;
- strengthening of charge transfer mechanisms;
- improved stability and controllability of the optical response.

These effects are particularly pronounced in GaSe/Cu intercalated heterostructures. The interaction between copper intercalants and irradiation-induced defects reduces the probability of carrier localization and increases the number of effective charge carriers. As a result, electrical conductivity is enhanced, the lifetime of photogenerated carriers is prolonged, and the optoelectronic response is strengthened. This synergistic effect clearly demonstrates the advantages of combining intercalation and irradiation strategies.

It should be emphasized that the results obtained using Co-60 gamma irradiation significantly enhance the prospects of heterostructures for radiation-resistant electronic and optoelectronic devices. Irradiation in the stimulative dose regime not only improves functional performance but also preserves stable operation in radiation-rich environments. This approach is of particular relevance for the development of devices intended for space technologies, nuclear energy systems, and environments characterized by high radiation backgrounds.

In summary, the application of Co-60 gamma irradiation to intercalated GaSe-based heterostructures should be regarded not merely as an auxiliary modification technique, but as an effective scientific tool for the fine and controlled tuning of functional properties. The stimulative dose regime selected by the author further demonstrates that this approach is both fundamentally sound and practically safe and efficient.

Industrial Advantages of the Intercalation Methodology

The intercalation methodology offers several significant advantages from an industrial perspective, making it one of the most promising technological approaches in heterostructure engineering. One of its primary advantages is the ability to carry out the process at relatively low temperatures. Compared to conventional methods such as high-temperature diffusion doping or ion implantation, this feature reduces energy consumption and prevents degradation of the crystal structure. During intercalation, the fundamental framework of the layered structure is preserved, and modifications occur predominantly within the van der Waals gap, thereby maintaining the mechanical integrity and structural stability of the material.

Another key advantage of intercalation lies in its scalability and compatibility with existing fabrication technologies. Its integration with chemical vapor deposition (CVD), physical vapor deposition (PVD), and various thin-film processing techniques enables a realistic transition from laboratory-scale studies to industrial manufacturing. In comparison with classical doping methods, intercalation induces significantly less structural damage, does not sharply increase defect density, and consequently preserves the crystalline quality and long-term functional stability of the material. Furthermore, the reversible nature of certain intercalation processes allows for stepwise and flexible tuning of functional parameters, which is of particular importance for the development of adaptive and “smart” devices.

GaSe-based intercalated heterostructures are of particular interest for industrial applications. This material platform is considered promising for high-sensitivity sensors, low-noise photodetectors, energy storage and conversion components, as well as flexible and transparent electronics. Through intercalation, the electrical conductivity and optical response of GaSe can be precisely tailored to meet specific application requirements. The studies conducted by the author demonstrate that such systems exhibit stable behavior under realistic operating conditions and provide a reliable scientific foundation for industry-oriented development. In this context, the intercalation methodology should be regarded not only as a tool for fundamental research but also as a practical and forward-looking approach for the commercialization of heterostructure-based technologies.

Comprehensive Enhancement of Functional Performance

The combination of intercalation and irradiation approaches enables a comprehensive and multiparametric enhancement of functional performance in heterostructure systems. While intercalation provides the initial modification of the interlayer space and electronic structure, irradiation acts as an external stimulus that further fine-tunes these changes. As a result of this synergy, electrical conductivity increases not only due to an enhanced carrier concentration, but also as a consequence of reduced scattering mechanisms and improved effective carrier mobility. In parallel, the passivation of recombination centers and the purposeful reorganization of defects extend the lifetime of photogenerated carriers and ensure greater stability of the optical response. Consequently, the intercalation–irradiation combination emerges as an integrated engineering strategy capable of simultaneously optimizing both electrical and optoelectronic parameters of heterostructures.

This conceptual approach is expected to play a significant role in the design of next-generation nanoelectronic and optoelectronic devices. In particular, the intercalation–irradiation synergy opens new opportunities for the development of devices that operate reliably in radiation-rich environments, maintain long-term stability, and exhibit tunable performance characteristics. Radiation-resistant field-effect transistors, high-sensitivity photodetectors, and photovoltaic elements with enhanced stability represent illustrative examples of potential application areas. From this perspective, the comprehensive enhancement of functional performance should be regarded not merely as the improvement of individual material parameters, but as a strategic approach that establishes a fundamental scientific basis for adapting heterostructure systems to real-world applications.

CONCLUSION

The conducted investigations demonstrate that the purposeful combination of intercalation and irradiation methodologies serves as an effective tool for the comprehensive regulation of electronic, optical, and structural properties in heterostructure systems. In particular, copper (Cu) intercalation in GaSe-based heterostructures exhibits significant advantages in enhancing electrical conductivity, strengthening charge transfer mechanisms, and ensuring functional stability. Furthermore, the application of a stimulative dose regime using a Co-60 gamma irradiation source enables material modification without inducing structural damage, while providing additional optimization of performance.

These findings indicate that the intercalation–irradiation synergy is not only of fundamental scientific interest but also of substantial practical relevance for real-world applications. GaSe-based intercalated heterostructures have been identified as a promising material platform for high-sensitivity sensors, radiation-resistant optoelectronic devices, and flexible electronics. The studies carried out by the author not only address existing knowledge gaps in this field but also establish a reliable scientific foundation for the industry-oriented advancement of heterostructure engineering. Ultimately, the approaches presented in this work open important prospects for the design and commercialization of next-generation functional materials.

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CHAPTER 4

WATERLESS PRODUCTION TECHNOLOGIES IN COSMETICS

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INTRODUCTION

The global cosmetics industry has grown rapidly over the last two decades, reaching a significant point in terms of both economic scale and product diversity. However, behind this growth lie critical sustainability problems such as high water consumption, energy requirements, packaging waste, and carbon emissions. The fact that the vast majority of cosmetic products contain water at rates ranging between 60% and 95% causes water to be used not only as a formulation component but also as a solvent, carrier, heat exchanger, and cleaning agent in the production line (Khan et al., 2023). The global decline in freshwater resources, increasing problems of access to water, and policies aimed at restricting water use in industries have made it necessary to reassess water in the cosmetics sector. In this context, the approach called "waterless cosmetics" or "anhydrous formulations" has come to the fore in terms of both sustainability and high-performance criteria.

The waterless cosmetics approach does not merely mean the absence of free water in the product. When evaluated within a broader framework, it refers to a holistic production paradigm that aims to minimize water use in formulation development, process design, packaging, storage, and supply chain processes (Lee & Park, 2021). The main reason for this is that water-based emulsions are prone to oxidation, suitable for microbial growth, and more fragile in terms of shelf life. In contrast, waterless formulations are increasingly preferred in both dermocosmetic and functional product groups because they have a low risk of microbial contamination, allow oxidation-sensitive actives to remain more stable, and enable high-concentration formulations.

The reduction of microbial growth potential as water activity (aw) decreases is one of the most important scientific bases of waterless formulations. A certain level of free water is required for microbial growth to occur. When the aw value falls below 0.6, proliferation ceases for most bacteria and yeast species (Roos, 2018). This feature significantly reduces the need for preservatives in waterless products, contributing both to the development of cleaner formulations and to the reduction of potential irritation risks. In addition, active substances that can degrade via hydrolysis-such as ascorbic acid, retinol, peptides, and AHA/BHA derivatives-remain much more stable in water-free systems, and this directly increases product efficacy (Chaudhary & Patel, 2022).

In recent years, the water-saving and concentrated cosmetics trend that started in Asian markets has also led to the strategic transformation of global brands. Water-free facial cleansers, oil-based serums, solid shampoos, and powder masks that emerged in Korea and Japan quickly became widespread in European and US markets. According to Euromonitor (2022) data, between 2015 and 2022, the waterless cosmetics market achieved an average annual growth rate of over 11%, and consumer preferences shifted markedly toward sustainability-focused products. This trend cannot be explained solely by environmental sensitivity; it is also associated with users' demand for higher active concentrations, more minimal ingredient lists, and longer shelf life.

A significant portion of the environmental burden created by the water used in cosmetics manufacturing stems not from the water directly added to the formulation, but from indirect water use. The amount of water used in CIP (Clean-in-Place) cleaning systems, cooling towers, distillation phases, and washing of process equipment is quite high when calculated per product. According to the UNEP (2023) report, the annual water footprint of the personal care industry is around 30–45 billion liters. This amount constitutes a critical pressure for manufacturers operating in regions where drought risk is increasing. The widespread adoption of waterless production technologies contributes to the long-term sustainability of the sector by reducing both water consumption and production costs.

Waterless production has not only environmental but also economic advantages. In liquid cosmetic products, because water creates volume and weight, packaging size, logistics costs, and storage space requirements increase. When solid shampoo bars are compared with liquid shampoos, it is reported that 7–10 times more product can be transported with the same production area and shipping capacity (Lövgren et al., 2021). This significantly reduces the carbon footprint and provides efficiency in the supply chain. In addition, the reduction of packaging waste constitutes an important motivation in terms of sustainable consumer behaviors.

The rise of waterless cosmetics has also created new research areas in formulation science. The development of solid surfactants, anhydrous solvents, wax–polymer networks, and nanotechnological encapsulation methods increases both the efficacy and the user experience of these products. For example, the use of mild surfactants such as SCI (Sodium Cocoyl Isethionate) in solid form offers advantages of low irritation potential and high biodegradability (Sarkar et al., 2022). However, technical challenges such as the solubility of some active substances, wax crystallization, rheological behavior, and stability are among the research topics in which waterless production technologies continue to progress.

It is also noteworthy that the number of domestic R&D companies in Türkiye that use water efficiently and develop waterless formulation technologies is increasing. Especially companies working in nanotechnology, polymer engineering, and sustainable chemical synthesis contribute both to reducing import dependency and to environmental impact management by increasing domestic production capacity. In this context, waterless cosmetics is not only a formulation trend but also a comprehensive transformation that represents the integration of circular economy approaches, green chemistry principles, and sustainable production models.

For all these reasons, the waterless production approach should be considered a scientific, technological, and environmental transformation shaping the future of the cosmetics sector. In this book chapter, the scientific foundations, sustainability impacts, formulation strategies, and industrial applications of waterless production technologies will be addressed within a broad literature framework.

CONCEPTUAL FRAMEWORK OF WATERLESS COSMETICS

Definition and Scope

Waterless cosmetics, in the narrow sense, is a concept used to define cosmetic products that do not contain free water in their formulation. However, in line with current scientific and industrial approaches, this definition has gone beyond a technical framework that focuses only on product content. Today, waterless cosmetics is evaluated as a holistic sustainability model encompassing raw material selection, formulation design, production technologies, cleaning processes, packaging, logistics, and product use habits (Lee & Park, 2021).

Traditional cosmetic products, especially in the skin care and personal cleansing segments, are based on a water phase that constitutes 60–90% of the formulation. Emulsion systems (oil-in-water or water-in-oil), gels, and lotions are structures dependent on water in terms of stability, spreadability, and sensory properties. However, the high proportion of water in the formulation increases the risk of microbial growth; this, in turn, makes the use of strong preservative systems mandatory. In addition, water creates an environment that increases the risk of degradation through hydrolysis and oxidation for many active ingredients (Roos, 2018).

The waterless cosmetics approach, with the removal of water from the formulation, not only increases the concentration of the product but also offers significant advantages in terms of product safety, shelf life, stability, and efficacy. Low water activity (aw) limits the proliferation of microorganisms, reduces the need for preservatives, and enables the development of products marketed as "clean beauty" or "preservative-free" (Chaudhary & Patel, 2022). In addition, the fact that heat- and moisture-sensitive actives remain more stable in water-free systems ensures that product performance is maintained for a long time.

The concept of waterless cosmetics also represents an important paradigm shift in terms of production processes. The high-volume water use common in traditional cosmetics production causes serious water and energy consumption through heating–cooling cycles, phase mixing operations, and especially CIP (Clean-in-Place) systems. In contrast, waterless production lines reduce environmental burden with less need for equipment washing, shorter process times, and low energy input. According to UNEP (2023) reports, process- and cleaning-related water consumption can be reduced by 50–70% in waterless production systems.

The fundamental elements defining waterless cosmetics are addressed under four main headings:

Absence of free water in the formulation

Although the absence of free water in the formulation does not mean that the product is completely free from microbial risk, low water activity seriously limits microbial growth. This makes it possible to reduce synthetic preservatives or completely remove them from the formulation. In addition, since pH dependency decreases, formulation flexibility increases and more tolerable products can be developed for sensitive skin (Chaudhary & Patel, 2022).

Minimization of water use in production processes

Waterless cosmetics is a process-based sustainability approach, not only content-based. Reducing water in production means reducing energy consumption, reducing the carbon footprint, and optimizing production costs in the long term. Especially in the production of solid and powder products, low temperature and pressure requirements further reduce environmental impact (UNEP, 2023).

Concentrated and high active ingredient content

Removing water allows active components in the formulation to be present at higher volumetric ratios. Actives such as vitamin C, retinoids, alpha and beta hydroxy acids, niacinamide, and peptides can remain stable for longer in waterless systems. Therefore, waterless products are generally positioned as "high performance," "highly concentrated," or "enhanced efficacy" (Sarkar et al., 2022).

Environmental advantages in packaging, storage, and logistics

Solid, powder, or oil-based waterless products can be packaged in smaller volumes and are generally offered with alternative packaging solutions that reduce plastic use. Transporting lighter products contributes to reducing logistics-related carbon emissions. In addition, long shelf life reduces energy consumption by eliminating the need for a cold chain (Lövgren et al., 2021).

When these elements are evaluated together, waterless cosmetics can be defined not only as a subfield of formulation science but as a multidimensional production and consumption model directly related to the climate crisis, water stress, and circular economy goals.

Product Types and Formulation Types

Waterless cosmetics encompasses a wide range of products with different physical forms and technological approaches. These products are examined under various categories according to the mode of use, targeted effect, and formulation technology.

Solid cosmetic products

Solid shampoos, soap bars, facial and body cleansing bars, solid lotions, and deodorant sticks are the main examples of this category. In these products, solid surfactants (SCI, SLSA, sodium cocoyl isethionate derivatives) and natural binders are generally used. The solid form both reduces packaging waste and offers the advantage of long-term use.

Anhydrous (waterless or containing water at a very low level) oil-based formulations

Oil serums, balm structures, ointments, and oil-based cleansers are included in this group. These systems provide advantages especially in carrying lipophilic actives and protecting components with high sensitivity to oxidation/hydrolysis. In addition, they are widely preferred in dermatological products due to their barrier-supporting effects.

Powder formulations

Clay masks, enzymatic peeling powders, ascorbic acid powders, and mineral-based makeup products constitute an important segment of waterless cosmetics. Activated by the user with water or hydrolate, these products offer the advantage of fresh use and long shelf life.

Concentrated tablet and compact systems

Cleansing tablets, dental care tablets, and compact products used by rehydration play an important role especially in reducing environmental impacts arising from packaging and logistics. These products offer solutions compatible with circular economy and refill systems.

Biotechnological and polymer-based systems

In next-generation waterless cosmetic products, biopolymer networks, oleogel systems, and nanostructured carriers that mimic the functions of water are used. These systems increase product efficacy by providing controlled release of actives (Wang et al., 2020).

This diversity clearly demonstrates that waterless cosmetics is not merely an alternative trend, but an innovative field located at the intersection of cosmetic science, materials engineering, and sustainable production technologies.

The Place of the Concept in the Cosmetics Sector

The waterless cosmetics approach is directly related to areas such as:

- sustainability trends,
- green chemistry principles,
- circular economy models,
- the minimal ingredient approach
- and high active stability.

In particular, the environmental sensitivity and minimal ingredient expectations of Generation Z consumers accelerate the growth of international markets. Euromonitor (2022) predicts that by 2030, the total market share of waterless cosmetics could rise to as much as 25%.

Historical Development

Although the concept of waterless cosmetics has become visible in the global market in recent years, its origins are based on much older practices. In the early periods of cosmetics history, a large part of the products used-such as clay masks, oil–wax mixtures, and pigment-based paints-were naturally waterless formulations. Malachite and coal powders used as eyeliner and eyeshadow in Ancient Egypt, vegetable oils used in skin care in Ancient Mesopotamia, and ointments prepared from wax and oil mixtures in the Roman period are accepted as historical examples of waterless cosmetics (Harper & Stone, 2019). Although the production techniques of these products were incomparably simple compared to modern technology, the limited use of water in cosmetic products was a necessity in those periods because water could not provide stability without requiring preservatives.

With the Industrial Revolution, the development of chemical science enabled the advancement of emulsion technology, and toward the end of the 19th century the production of water-based cosmetics began to become widespread. Especially from the mid-20th century onward, the ability to produce O/W (oil–water) emulsions at industrial scale fundamentally changed the scientific and commercial structure of cosmetics. The widespread adoption of emulsions standardized the use of water as the main component in products such as creams, lotions, shampoos, and cleansers. This period was also a process in which preservative technologies developed, product stability increased, and cosmetics became more refined (Löwenthal, 2008). However, these developments also brought the environmental impacts of high water use in the long term.

The modern emergence of the concept of waterless cosmetics dates back to the early 2000s. Minimalist care routines, concentrated serum technologies, and solid cleansing products that developed in Japan and South Korea formed the foundations of new formulation approaches that reduce water use. In this period, the K-Beauty and J-Beauty movements rapidly increased interest in products that reduce water use because they emphasized both ingredient simplification and environmental sustainability. Lee and Park (2021) state that one of the main reasons this trend was born in Asia is local consumers' interest in formulations with high active content and low preservatives.

Between 2010 and 2015, environmental sustainability debates intensified globally; concepts such as water scarcity, energy efficiency, carbon footprint, and waste management became key regulatory focuses for the cosmetics industry. Among the Sustainable Development Goals published by the United Nations in 2015 were items such as protecting water resources, increasing water efficiency in production processes, and reducing industrial consumption; this increased the strategic importance of production models with low dependence on water (UNEP, 2015). These developments directed the R&D teams of cosmetics companies toward work on developing waterless formulations.

The global acceleration of waterless cosmetics occurred especially after 2015. According to Euromonitor (2022) data, between 2015 and 2022 the average annual growth rate of the waterless cosmetics market rose above 11%; solid shampoos, oil-based serums, powder cleansers, and concentrated masks were the fastest-growing categories. During this period, many international brands added waterless series to their product portfolios, taking steps aligned with environmental sustainability goals and responding to consumers' expectations for minimal ingredients and high performance.

Technological developments also accelerated this transformation. In the post-2018 period, innovations such as encapsulation technologies, the development of solid surfactants, the synthesis of polymer networks replacing solvents, and nanostructures capable of carrying actives in the oil phase took waterless cosmetics to a much more advanced level than simple solid products (Wang et al., 2020). Thanks to these technological advances, it has become possible to protect sensitive actives such as vitamin C, peptides, and retinoids more stably without contact with water.

Today, waterless cosmetics is considered one of the most innovative areas of the cosmetics industry in terms of both environmental sustainability and formulation science. In the modern sense, waterless cosmetics is a strategic innovation area shaping the future of the sector due to its multidimensional benefits such as:

- ensuring microbial stability by reducing water activity,
- reducing the need for preservatives,

- reducing transport and packaging burden,
- minimizing energy consumption,
- increasing the stability of high-concentration actives.

In addition, being located at the intersection point of global trends such as circular economy, low carbon footprint, minimal ingredients, and the clean cosmetics movement accelerates moving away from water-dependent production models. For manufacturers, this area is not only a market opportunity but also a critical strategic tool in terms of regulatory compliance, reducing environmental impacts, and strengthening brand value.

As a result, the historical development of waterless cosmetics can be considered an evolutionary process extending from the simple oil–wax mixtures of ancient civilizations to modern nanotechnological formulations. This development has been shaped by the combination of scientific advances and environmental, economic, and sociocultural factors. Today, waterless cosmetics is seen as a strong approach at the center of sustainable production strategies.

ENVIRONMENTAL IMPACTS OF WATER USE IN COSMETICS

The cosmetics industry is one of the sectors that has been rapidly growing on a global scale and, in parallel, increasing natural resource consumption. In this sector, water plays a critical role not only as a basic component of product formulations but also in many processes such as production, cleaning, cooling, energy production, and waste management (UNEP, 2023). A large part of traditional cosmetic products contains water at a rate of 60–90%, which significantly increases both direct and indirect water consumption (Lövgren et al., 2021). Therefore, the environmental impacts of water use in cosmetics should be addressed not only as limited to product content but as a multidimensional sustainability issue encompassing the entire production and supply chain.

In cosmetic production facilities, water consumption occurs intensively for the preparation of raw materials, mixing, heating and cooling operations, as well as for ensuring the cleaning and hygiene of production lines. In particular, cleaning lines and CIP (Clean-in-Place) systems require the use of high amounts of water and chemical cleaning agents, and this constitutes a significant part of the total water footprint of production facilities (UNEP, 2023). In addition, cooling towers, steam boilers, and energy production processes are among the factors that increase indirect water consumption.

However, the environmental impact of water in the cosmetics sector is not limited only to the amount consumed. The processes of treating, transporting, and discharging the water used also create significant environmental burdens. Especially insufficient treatment of wastewater containing surfactants, preservative systems, and synthetic components can lead to toxic effects in water ecosystems and a decrease in biodiversity (UNEP, 2023). In this context, reducing water use in cosmetics is of critical importance both for resource efficiency and ecosystem health.

Water Footprint

The water footprint is a holistic indicator referring to the total amount of freshwater consumed directly and indirectly throughout the life cycle of a product or service. In terms of the cosmetics industry, this concept encompasses stages such as raw material production, formulation, manufacturing, packaging, distribution, and post-use phases (UNEP, 2023). The annual water footprint of the global cosmetics industry is estimated to be approximately 30–45 billion liters (UNEP, 2023).

A significant portion of the water footprint stems not from the water used in the formulation but from indirect processes. Agricultural irrigation used in the production of vegetable oils, extracts, and natural active ingredients significantly increases the total water footprint of cosmetic products (Lövgren et al., 2021). In addition, the production of plastic, glass, and paper-based packaging raw materials also requires the consumption of high amounts of water. This demonstrates that the environmental impact of cosmetic products cannot be evaluated solely by the content of the final product.

For manufacturers operating in regions under water scarcity risk, the water footprint has become a strategic risk factor. Climate change, increasing population, and irregular precipitation regimes make access to water a fragile issue both environmentally and economically (UNEP, 2023). In this context, the development of waterless or low-water-content cosmetic products stands out as an effective approach in reducing the water footprint.

Impacts on Carbon Footprint

Water use in the cosmetics sector is directly related to the carbon footprint. The processes of sourcing, treating, transporting, storing, and heating water all require high energy consumption, and when this energy is supplied mainly from fossil fuels, a significant amount of greenhouse gas emissions occurs (Lövgren et al., 2021). Therefore, reducing water consumption also contributes to lowering carbon emissions.

Traditional liquid cosmetic products, due to their high water content, create more weight and volume in transportation and logistics stages. This increases fuel consumption and carbon emissions per product. Solid shampoos and concentrated product forms, on the other hand, can be 80–90% lighter compared to traditional products and thus significantly reduce logistics-related emissions (Lövgren et al., 2021).

Waterless formulations also provide energy efficiency in the production stage. In water-free products, the need for heating and cooling decreases, less preservative system is required for microbial stability, and production processes become simplified. In addition, the use of smaller and lighter packaging reduces both raw material consumption and the amount of packaging waste. According to life cycle assessment (LCA) studies, it is reported that waterless cosmetic products can reduce the total carbon footprint by 20–65% (Lövgren et al., 2021).

As a result, reducing water use in cosmetics provides a holistic sustainability approach that enables the reduction of the carbon footprint together with the water footprint. Waterless cosmetics strategies provide important advantages not only in terms of environmental benefits but also in terms of cost reduction, supply chain resilience, and brand sustainability (UNEP, 2023; Lövgren et al., 2021).

WATERLESS PRODUCTION TECHNOLOGIES

In the cosmetics industry, waterless production technologies are among the most innovative approaches developed in line with sustainability goals. In traditional cosmetics production, although water is used only as a carrier medium, it has been accepted as an indispensable component for reasons such as formulation stability, microbial control, and process convenience. However, increasing environmental awareness, water scarcity risks, and global targets to reduce carbon footprint have made it necessary to reassess the role of water in the cosmetics sector. In this context, waterless production technologies not only reduce environmental impact but also offer advanced formulation strategies that increase product performance and functionality (UNEP, 2023).

Waterless production approaches encompass different technological platforms such as solid surfactant systems, wax–oil-based structures, powder cosmetics technologies, encapsulation strategies, and cold process methods. The common feature of these systems is that free water is completely or largely removed from the formulation and active ingredients are presented in a more concentrated, stable, and efficient structure.

Shampoo and Cleansing Systems with Solid Surfactants

One of the most common application areas of waterless production technologies is solid surfactant systems used in shampoo and cleansing products. Surfactants such as Sodium Cocoyl Isethionate (SCI), Sodium Coco Sulfate (SCS), and Sodium Lauryl Sulfoacetate (SLSA) can be easily integrated into water-free formulations thanks to their powder or granular forms. These surfactants have much higher active matter ratios compared to soluble surfactants used in traditional liquid cleansers and generally provide active content in the range of 70–85% (Barel et al., 2019).

One of the most important advantages of solid surfactant-based shampoos and cleansers is that transportation and storage costs per product are significantly reduced. Thanks to the high active concentration, the same washing performance can be achieved with smaller product volumes, which contributes to lowering carbon emissions in logistics processes (Lövgren et al., 2021). In addition, isethionate-based surfactants such as SCI are widely preferred in sensitive skin and scalp products due to their low irritation potential. This shows that waterless formulations are advantageous not only environmentally but also dermatologically.

Heat-Sensitive Wax Systems

In the development of cream, ointment, and stick formulations in waterless cosmetic products, wax–oil-based systems have an important place. In these systems, instead of the carrier role of water, vegetable or synthetic oils and waxes with different melting points are used. Wax systems provide structure to the formulation and ensure the physical stability of the product (Rastogi & Pandey, 2018).

In the design of heat-sensitive wax systems, the type and ratio of wax are of critical importance. Beeswax, carnauba wax, candelilla wax, and synthetic wax derivatives offer different hardness and melting profiles, determining the spreadability and sensory properties of the product. In waterless systems, wax-based structures also reduce the need for preservatives because they largely eliminate the risk of microbial growth. This stands out as an important advantage especially for natural and "clean beauty" concept products (UNEP, 2023).

Powder Cosmetics Technologies

Powder cosmetics technologies are another important component of waterless production approaches. Foundations, face masks, peeling products, and serum–powder mixtures are developed as water-free systems enabling high active loading. In these products, active ingredients are activated before use with water or an appropriate carrier, making it possible to obtain a fresh formulation (Barel et al., 2019).

One of the most important advantages of powder formulations is long shelf life and high chemical stability. Since they do not contain water, stability problems such as oxidation, hydrolysis, and microbial degradation are largely eliminated. In addition, offering dosage and usage flexibility to the user also enables personalized cosmetic applications. These features place powder cosmetics technologies at the center of the waterless production paradigm (Lövgren et al., 2021).

Microencapsulation and Encapsulation Technologies

In waterless production systems, microencapsulation and encapsulation technologies play a critical role especially in stabilizing hygroscopic and chemically unstable active ingredients. Niacinamide, alpha hydroxy acids (AHAs), and some vitamin derivatives are components that can rapidly degrade or become reactive in water-containing systems. Stabilizing these actives in waterless bases through lipid-based encapsulation methods offers significant advantages in terms of efficacy and shelf life (Wang et al., 2020).

Lipid-based encapsulation systems enable controlled release of active ingredients, reducing irritation risk on the skin and increasing bioavailability. In addition, encapsulation improves the performance of waterless formulations significantly by protecting active ingredients from environmental factors (Wang et al., 2020). Therefore, microencapsulation technologies are considered among the advanced engineering solutions of waterless cosmetics production.

Cold Process Technologies

One of the important advantages of waterless production technologies is that they allow cold process methods. In water-free systems, emulsion formation or phase transitions requiring high temperatures are generally not necessary. This reduces the need for thermal treatment during production and lowers energy consumption (UNEP, 2023).

Cold process technologies provide advantages both in energy efficiency and in protecting heat-sensitive active ingredients. Vitamins, enzymes, and biological actives can be included in the formulation without exposure to high temperatures and can maintain their efficacy for longer. In addition, the simplification of the production process shortens process times and provides cost advantages at industrial scale (Lövgren et al., 2021).

As a result, waterless production technologies provide multidimensional contributions to the cosmetics industry in terms of environmental sustainability, product performance, and production efficiency. Solid surfactants, wax systems, powder formulations, encapsulation technologies, and cold process approaches constitute the technological infrastructure of waterless cosmetics and shape the future of the sector.

FORMULATION SCIENCE

The success of waterless cosmetic products is possible not only by removing water from the formulation but also by properly designing the physicochemical properties of the components used in its place. In water-free systems, formulation science, unlike classical emulsion-based approaches, requires precise control of oil phase interactions, wax crystallization, active ingredient solubility, and rheological behaviors. Therefore, waterless cosmetic formulations are considered functional systems that require a high engineering approach (Barel et al., 2019).

The main goals in formulation design are to ensure active ingredient stability, obtain desired sensory properties, extend shelf life, and maintain product performance in a sustainable manner. Especially in dermocosmetic products containing actives sensitive to oxidation and hydrolysis, waterless systems offer significant advantages in achieving these goals (UNEP, 2023).

Basic Components

The basic components used in waterless cosmetic formulations carry a much higher functional load than water-based systems. The selection and ratios of these components are directly decisive on the stability and performance of the product.

Vegetable oils constitute the main carrier phase of waterless formulations. Oils with an unsaturated fatty acid profile such as jojoba oil and grape seed oil are widely preferred thanks to their biocompatible structures with the skin and their oxidative stability. While jojoba oil shows sebum-like properties due to its liquid wax ester structure, grape seed oil provides advantages in dermocosmetic applications with its light texture and antioxidant content (Barel et al., 2019).

Waxes are the main components that ensure structural integrity in waterless systems. Vegetable waxes such as candelilla and carnauba determine the hardness, spreadability, and stability of the product thanks to their different melting points and crystal structures. These waxes increase the physical durability of the product especially in stick, balm, and ointment formulations while also contributing to controlled release properties (Rastogi & Pandey, 2018).

Powder vitamins and actives play a critical role in maintaining stability in waterless formulations. Components such as ascorbic acid, tocopherol, and niacinamide can rapidly degrade in water-containing systems, whereas they exhibit a much longer shelf life in waterless matrices. This stands out as an important factor that increases the dermocosmetic efficacy of waterless formulations (Wang et al., 2020).

Anhydrous solvents are functional liquids used to ensure the homogeneous distribution of active ingredients. Solvents such as propanediol and isododecane are preferred due to both their low volatility and their ability to leave a light and silky feel on the skin. In addition, these solvents contribute to product efficacy by increasing skin penetration of actives (Barel et al., 2019).

Polymer network structures and especially crosspolymer systems play an important role in rheological control and stability in waterless cosmetics. These polymers prevent phase separation by forming three-dimensional network structures within the oil phase and support the long-term physical stability of the product (UNEP, 2023).

Example Waterless Formulation: Dermocosmetic Balm

As an applied example of waterless formulation science, a dermocosmetic balm formulation is presented below:

- 30% Shea butter
- 20% Jojoba oil
- 10% Candelilla wax
- 5% Tocopherol
- 2% Encapsulated Vitamin C
- 0.5% Fragrance
- q.s. Active complex

This formulation exhibits a structure that offers high stability especially for active ingredients sensitive to oxidation. The combination of shea butter and jojoba oil provides both barrier-repairing and moisturizing effects, while candelilla wax gives the formulation sufficient hardness and structural strength (Rastogi & Pandey, 2018).

Tocopherol functions in the formulation not only as an active ingredient but also as a fundamental element of the antioxidant system. In this way, oxidative degradation in the oil phase is prevented and shelf life is extended (Barel et al., 2019). Encapsulated vitamin C is stabilized within the lipid matrix to provide controlled release and reduce the risk of irritation on the skin (Wang et al., 2020).

Since such waterless balm systems do not carry a risk of microbial growth, they minimize the need for preservatives and offer a safe product profile especially for sensitive skin. In addition, thanks to the concentrated structure, the packaging and transport burden per product is also reduced (UNEP, 2023).

Rheology and Structural Behavior

In waterless cosmetic formulations, rheological behavior is one of the fundamental parameters that directly affects product performance and consumer experience. In solid and semi-solid systems, flow behavior deviates from classical Newtonian models and mostly shows plastic or pseudoplastic character. In this context, the Casson model and the Bingham plasticity model are widely used in rheological analysis of balm and stick formulations (Barnes, 2000).

The Casson model is particularly important in determining yield stress at low shear rates and provides critical information about the product's خروج from the packaging and applicability to the skin. Bingham plasticity defines the initiation of flow when a certain threshold stress is exceeded and is used to explain the structural strength of balm systems (Barnes, 2000).

In addition, wax crystallization kinetics plays a decisive role in the long-term stability of waterless systems. The size, distribution, and recrystallization tendency of wax crystals can lead to physical instabilities such as hardening over time or oil leakage. Therefore, cooling rate, mixing profile, and wax ratios must be carefully optimized (Rastogi & Pandey, 2018).

As a result, in waterless cosmetic formulations, rheology should be evaluated as a strategic design parameter not only for fluidity but also in terms of product stability, sensory perception, and user satisfaction.

INDUSTRIAL SCALE AND PROCESS DESIGN

Transferring waterless cosmetic products from laboratory scale to industrial production requires different engineering approaches compared to classical water-based cosmetic processes. Water-free or low water activity systems present unique process challenges in terms of high viscosity, phase continuity, and thermal behavior. Therefore, in the industrial scaling process, equipment selection, mixing geometry, thermal control, and cleaning strategies should be addressed with a holistic design approach (Barel et al., 2019).

Industrial-scale waterless production processes not only increase environmental sustainability but also offer significant advantages in terms of production efficiency, energy optimization, and consistency of product quality. However, obtaining these advantages is possible by correctly defining process parameters and configuring the production line in accordance with waterless systems (UNEP, 2023).

Mixer and Equipment Selection

In the production of waterless cosmetic formulations, mixer and reactor selection is one of the most critical factors determining process success. Low water activity systems generally have high viscosity, and traditional low-shear mixers are insufficient to obtain a homogeneous mixture. Therefore, high-shear mixers, planetary mixers, and twin-shaft mixers are widely used in waterless production (Barnes, 2000).

High shear force ensures effective distribution of wax and oil phases, homogeneous dispersion of active ingredients, and stable formation of polymer network structures. In particular, the ability to incorporate encapsulated actives and powder components into the system without forming agglomeration depends on the selection of an appropriate mixing geometry (Wang et al., 2020).

Jacketed reactors provide important advantages in terms of thermal control in waterless systems. Controlled execution of melting and recrystallization processes in wax-based formulations directly affects the rheological properties and physical stability of the product. With jacketed systems, homogeneous heat distribution is provided and local overheating is prevented (Rastogi & Pandey, 2018).

In addition, vacuum melting tanks are widely preferred in waterless cosmetics production. Melting and mixing operations carried out under vacuum remove air bubbles from the system, reducing oxidative degradation and improving the visual quality of the product. At the same time, vacuum systems allow controlled preservation of volatile components (Barel et al., 2019).

In industrial-scale equipment selection, factors such as surface roughness, ease of cleaning, and material compatibility should also be taken into consideration. Stainless steel (AISI 316L) surfaces are accepted as the standard for waterless cosmetics production in terms of both chemical resistance and hygiene (ISO 22716).

Water Consumption in CIP Systems and Process Optimization

In cosmetics production facilities, cleaning processes constitute a significant portion of total water consumption. In traditional water-based cosmetics production, CIP (Clean-in-Place) systems consist of many stages such as pre-rinse, detergent washing, intermediate rinse, disinfection, and final rinse, and require the use of high amounts of water (ISO 22716).

One of the most important environmental advantages of waterless production technologies is water savings achieved in CIP processes. In water-free or low water activity systems, product residues are less sticky and the risk of microbial contamination is significantly reduced. This allows cleaning steps to be simplified and the need for rinsing to be reduced (UNEP, 2023).

Industrial reports published within the scope of ISO 22716 show that, in facilities producing waterless products, an average of 60–70% water savings can be achieved in CIP steps (ISO 22716). In addition, the decrease in the amount of detergent and disinfectant used significantly reduces the wastewater treatment load. This contributes not only to conserving water resources but also to reducing chemical consumption and operating costs.

In terms of process optimization, pre-cleaning methods such as dry cleaning and mechanical scraping are also widely used in waterless systems. These approaches minimize the need for water and chemicals by ensuring that most of the product residues are physically removed before CIP (Barel et al., 2019).

In addition, automation-supported CIP systems increase both hygiene standards and resource efficiency by enabling precise control of cleaning time, water temperature, and flow rate. CIP scenarios specially designed for waterless production lines reduce production interruptions and support operational sustainability throughout the facility (UNEP, 2023).

Challenges in Industrial Scaling and Strategic Approaches

Although waterless cosmetics production offers significant advantages, some technical challenges are also present in the industrial scaling process. Pumping high-viscosity systems, ensuring flow stability on filling lines, and managing temperature control sensitively are among the main challenges. Therefore, conducting pilot-scale studies and carefully determining scale-up coefficients are of great importance in process design (Barnes, 2000).

On the other hand, with appropriate equipment selection, optimization of process parameters, and personnel training, these challenges can be effectively managed. Industrial-scale waterless production offers a strategic competitive advantage to the cosmetics sector in the long term in terms of both environmental and economic sustainability.

RESEARCH AND INNOVATION AREAS

Waterless cosmetics production stands out as a dynamic field that brings advanced research and innovation activities along with it, rather than being limited to developing water-free versions of existing formulations. Increasing environmental pressures, regulations, and consumer expectations direct cosmetic R&D studies toward more sustainable, high-performance, and technology-focused solutions. In this context, waterless cosmetics systems are shaped by interdisciplinary approaches such as biotechnology, nanotechnology, polymer science, and controlled release engineering (UNEP, 2023).

Current research shows that waterless formulations not only provide environmental benefits but also offer significant advantages in terms of active ingredient stability, targeted effect, and product lifetime. Therefore, waterless production has started to take a central place in innovative product development strategies in the cosmetics sector (Lövgren et al., 2021).

Enzymatic Powder Formulations

Enzymatic actives attract great interest in cosmetic applications due to their high biological activity, but they experience stability problems in water-containing systems. Hydrolysis, microbial degradation, and loss of activity are the main factors limiting the shelf life of enzyme-based products. Therefore, developing enzymes in the form of waterless or low-moisture powder formulations has become an important research area in recent years (Barel et al., 2019).

Enzymatic powder formulations increase stability and maximize targeted biological activity with "fresh-mix" concepts that are activated at the time of use. Proteases, lipases, and antioxidant enzymes can be integrated into cosmetic products in a more controlled and safe manner thanks to this approach. In addition, water-free environments allow enzymes to remain active for a longer time in the carrier matrix (UNEP, 2023).

Nanotechnological Active Carriers

Nanotechnology is one of the most important innovation areas that increase the efficacy of waterless cosmetic formulations. Nanoemulsions, nanolipid carriers, and polymeric nanoparticles significantly improve the solubility, skin penetration, and bioavailability of active ingredients (Wang et al., 2020).

The use of nanotechnological carriers in waterless systems offers critical advantages especially in protecting actives sensitive to oxidation and light. Active ingredients are physically and chemically isolated within nanostructures, protected against environmental factors, and controlled release is achieved in the target tissue. This both increases product performance and reduces irritation risk (Lövgren et al., 2021).

In addition, nanotechnological systems allow high impact at low dose, optimizing raw material use. This approach is also considered a strategic innovation area in terms of sustainability and cost effectiveness.

Kinetically Controlled Release Systems

Kinetically controlled release systems are among the advanced engineering solutions that increase the functional value of waterless cosmetic formulations. In these systems, the release of active ingredients is performed in a controlled manner through diffusion, matrix degradation, or environmental triggers (temperature, pH, mechanical stress) (Wang et al., 2020).

Especially in dermocosmetic products targeted for long-term effect, controlled release systems ensure that the active ingredient shows a more balanced and continuous effect on the skin. Waterless matrix structures allow such kinetic control mechanisms to be designed in a more predictable and stable way. This approach increases product efficacy while also supporting user safety (Barel et al., 2019).

Waterless Bio-Based Polymers

A significant part of sustainability-focused research consists of developing bio-based polymers suitable for waterless systems. Plant-derived or biodegradable polymers developed as alternatives to petroleum-derived polymers are used in waterless cosmetic formulations to optimize rheological control, stability, and sensory properties (UNEP, 2023).

These polymers both reduce environmental impact and contribute to preventing environmental risks stemming from microplastics. Waterless bio-based polymer systems are considered a strategic research area in the future in terms of regulatory compliance and creating sustainable product portfolios.

CONCLUSION

Waterless cosmetics is an innovative production approach that offers a comprehensive solution to the environmental, economic, and technological problems faced by today’s cosmetics industry. In a period when fundamental problems such as global water scarcity, the necessity of reducing carbon footprint, energy consumption, packaging waste, and supply chain inefficiencies are intensifying, developing formulations that minimize water use has increasingly become a strategic necessity. In this context, waterless cosmetics should be considered not only a new formulation trend but also a transformation movement integrating sustainable production, green chemistry principles, and the circular economy understanding.

The most fundamental advantage of waterless cosmetics systems is that removing water from the formulation increases the stability of actives, reduces the risk of microbial degradation, and minimizes the need for preservatives. Low water activity limits the environment required for microorganisms to multiply; this significantly improves both product safety and shelf life (Roos, 2018). For example, actives sensitive to oxidation and hydrolysis such as ascorbic acid, peptides, and retinoids can remain stable for much longer in a waterless structure and show high efficacy. This feature brings waterless products to the fore especially in the categories of dermocosmetics, anti-aging, and high-performance care products.

Another important advantage of the waterless cosmetics approach is that it enables high active concentration. The fact that most of the formulation consists of oils, waxes, polymers, or powder actives instead of water has enabled the product to have a more minimal ingredient list and has achieved strong compatibility with the “clean beauty” trend. Consumers’ increasing level of awareness and preference for short ingredient lists with fewer preservatives and synthetic additives increase the market attractiveness of waterless cosmetics (Sarkar et al., 2022).

From the perspective of environmental sustainability, waterless cosmetics is critically important in reducing the total water footprint of the industry. The amount of water used in cosmetics production is not limited only to the water in the formulation; high amounts of water are also used in areas such as CIP cleaning systems, washing of process equipment, solvent recovery processes, and cooling water (Khan et al., 2023). Waterless formulations have lower energy requirements compared to classical emulsions and include fewer heating–cooling cycles, thereby reducing the carbon footprint of production facilities. In addition, because these products generally need lighter and more compact packaging, they can reduce carbon emissions per transported product by 20–60% in logistics processes (Lövgren et al., 2021).

When evaluated in terms of the supply chain, the fact that waterless cosmetics have low storage, transport, and shelf space requirements offers economic advantages for both manufacturers and retailers. Products such as solid shampoo bars, compact cleansing tablets, and powder formulations make it possible for more units of product to fit into a smaller volume; thus, logistics capacity efficiency increases significantly. This creates a cost-reducing effect especially for brands with high export volumes.

However, the waterless cosmetics approach also has some technical and application-oriented limitations. These include technical problems such as wax crystallization behavior, oil phase rheology, dissolution rate of solid surfactants, the user adaptation process to texture and usage experience, and the insufficient solubility of some actives in the oil or powder phase. In addition, for consumers accustomed to the usage comfort of water-based products, the practicality of applying waterless products can sometimes create difficulties. Therefore, studies on formulation engineering, sensory analysis, and user habits remain important in the field of waterless cosmetics.

Looking at future project and research areas, the potential of waterless cosmetics is quite broad. Nanotechnological active carriers, polymeric network structures that can replace water, biotechnological solvents, enzymatic powder technologies, and heat-sensitive gelation systems constitute important R&D opportunities in this field (Wang et al., 2020). Especially the development of biopolymers based on sustainable raw materials will make waterless formulations stronger both environmentally and in terms of performance. At the same time, in line with circular economy principles, refillable, minimum-packaging, or packaging-free product designs will be one of the main application areas of the future.

From a sectoral perspective, waterless cosmetics is not only a product category but also a transformation tool affecting the entire production process. The R&D units, supply chain managers, and production engineers of companies must manage this transformation together. Moreover, consumer expectations, sustainability reporting obligations, and international environmental policies stand out as external factors accelerating this transformation. Therefore, waterless cosmetics is a candidate to be an important component not only of the cosmetics sector but also of the future of sustainable production models.

As a result, waterless cosmetics offers a scientifically grounded, environmentally sustainable, economically advantageous, and technologically innovative approach. This approach both produces solutions to current environmental problems and introduces new design paradigms in cosmetic formulations. In a world where water resources are increasingly decreasing, developing waterless cosmetics technologies is not just a trend but also a mandatory production model for future generations. Investments the sector will make in this field will facilitate reaching sustainability goals and add a new dimension to the scientific development of cosmetics.

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CHAPTER 5

ZERO-WASTE AND CARBON-NEGATIVE PROCESS DESIGN IN THE FOOD INDUSTRY

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INTRODUCTION

The food industry operates under significant environmental pressures due to the growing global population, changing consumption habits and the climate crisis. The food production chain is considered one of the main sources of greenhouse gas emissions, characterised by high energy and water consumption, significant food loss, and waste generation. For example, food waste leads to both economic losses and increased environmental burdens, making waste management and carbon footprint critical issues in the food industry (Sudarsan et al., 2025).

FAO and sustainability literature reveal that approximately one-third of the food produced worldwide is lost or wasted during production, processing, and distribution, a situation which leads not only to economic, but also environmental costs (e.g. energy consumption and greenhouse gas emissions) (Suthiluk et al., 2025). In this context, the need to reevaluate current production paradigms considering environmental sustainability criteria has become the focus of academic and industrial research.

In response to this need for transformation, the concepts of zero-waste and carbon-negative production are gaining increasing importance in food engineering literature. While the zero-waste approach aims to reduce or eliminate waste in processes, carbon-negative strategies aim not only to reduce carbon emissions, but also to achieve net carbon removal. For example, some studies show that biochemical waste valuation and integrated system approaches can address both waste reduction and carbon footprint estimation simultaneously (Shabir et al., 2023).

From a food engineering perspective, achieving zero-waste and carbon-negative goals is not limited to technological innovations, but is also possible through engineering strategies such as system-level process integration and optimization of energy and material flows. In this context, concepts such as process integration, energy recovery, water reuse, and industrial symbiosis have become key.

The Concept of Zero-Waste in Food Processes

The concept of zero-waste is a holistic sustainability approach that focuses on preventing waste generation in production systems from the outset and on reusing, recycling, or converting unavoidable by-products into high-value-added products. From the perspective of the food industry, the zero-waste concept requires a systematic process design that encompasses not only final product losses but also all material, energy, and water losses that occur during raw material preparation, processing, storage, and packaging (Ghisellini et al., 2016).

Traditional food production systems are mostly based on the linear 'take–make–use–dispose' model. In this model, by-products and waste are generally considered burdens to be disposed of, which leads to unsustainable results both environmentally and economically. In contrast, the zero-waste approach in food processes aims to abandon the linear model and instead design closed-loop systems based on circular economy principles. In circular production systems, waste serves as raw material for another process, which significantly increases resource efficiency (Geissdoerfer et al., 2017).

Waste and by-product formation is common in food processing. In the fruit and vegetable processing industry, peels, pulp, and seeds are produced in large quantities, while in the dairy industry whey is produced, and in grain processing bran is produced, and in oilseed processing, meal and pulp are also produced. The literature clearly shows that these by-products are rich in carbohydrates, proteins, dietary fiber, and phenolic compounds, and that they can be converted into functional food components or industrial raw materials with appropriate engineering approaches (Mirabella et al., 2014; Galanakis, 2020).

One of the cornerstones of the zero-waste approach is the waste hierarchy. This offers a multi-layered system including waste prevention, reduction, reuse, recycling and, as a last resort, energy recovery. From a food engineering perspective, the first step in this hierarchy, waste prevention, can be achieved by optimizing process parameters, increasing raw material efficiency, and minimizing process losses. Therefore, process design and optimization play a critical role in achieving zero-waste goals (Papargyropoulou et al., 2014).

Process integration and industrial symbiosis approaches are of great importance for the effective implementation of zero-waste practices in food processes, which are vital in today's world. Industrial symbiosis enables the by-products generated by one business to be used as raw materials by another business or in another process, thereby increasing resource efficiency on a regional scale. It has been reported that symbiotic systems implemented in food production clusters significantly reduce waste quantities and minimize environmental impacts (Chertow, 2000; Lombardi and Laybourn, 2012).

Recent studies show that adopting zero-waste principles in the food industry provides environmental benefits and positively impacts the economic performance of businesses. Reduced waste disposal costs, the commercialization of by-products and decreased energy and water consumption are among the key factors that increase the industry's profitability.

Carbon Footprint and Carbon-Negative Approaches in The Food Industry

The carbon footprint of a product or system is the total amount of greenhouse gases emitted directly and indirectly throughout its life cycle, and is usually reported in carbon dioxide equivalents (CO_2-equivalents). The food industry is responsible for a significant portion of global greenhouse gas emissions due to its complex supply chain structure, which extends from agricultural production to industrial processing, transportation, and storage. Current estimates suggest that 26–34% of global greenhouse gas emissions are linked to food systems (Vermeulen et al., 2012; IPCC, 2023).

Although the food processing stage accounts for a lower share of total food system emissions compared to agricultural production, it constitutes a critical component of the carbon footprint due to energy-intensive unit operations. Processes such as thermal treatment, drying, evaporation, cooling and freezing result in high electricity and fossil fuel consumption. Additionally, process water usage, wastewater treatment, and auxiliary operations (steam production, cooling systems) increase indirect greenhouse gas emissions (Jeswani et al., 2015; Garnett, 2011). Therefore, energy efficiency in the design and operation stages of food processes plays a decisive role in reducing the carbon footprint.

The most used method for quantitatively assessing the carbon footprint in the food industry is Life Cycle Assessment (LCA). The LCA approach, within the framework of ISO 14040 and ISO 14044 standards, enables the systematic analysis of all environmental impacts of a product from "cradle to grave" or "cradle to door." LCA studies in food processes reveal the comparative effects of energy sources, raw material inputs, by-product flows, and waste management strategies on the carbon footprint (ISO, 2006a; ISO, 2006b).

In recent years, the concept of carbon-negative production, which goes beyond reducing the carbon footprint, has been increasingly discussed in the literature. Carbon-negative systems aim not only to minimize greenhouse gas emissions but also to achieve net carbon removal from the atmosphere. In the context of the food industry, this approach requires the combined consideration of renewable energy integration, biochar production from waste and by-products, carbon capture and utilization (CCU), and biological carbon sequestration mechanisms (Lehmann and Joseph, 2015; Minx et al., 2018).

Biochar, which is derived from food processing by-products, offers significant potential among carbon-negative strategies. Biochar is produced through the pyrolysis of biomass in an oxygen-free environment and contributes to the permanent sequestration of atmospheric carbon due to its long-term carbon storage capacity. It is also considered a strategic tool for closed-loop carbon management in food systems due to its soil-improving properties and positive effects on agricultural productivity (Lehmann et al., 2011; Woolf et al., 2010).

However, technology selection alone is not sufficient for designing carbon-negative food processes. We must optimize energy and material flows at the system level, integrate processes, and convert waste into high value-added products. For example, waste heat recovery, biogas production and integration with renewable energy systems can significantly improve the net carbon balance of food factories. Such integrated approaches enable the food industry to go beyond carbon reduction targets and evolve towards carbon-negative production models (Garnett, 2011; Poore and Nemecek, 2018).

Consequently, the concept of carbon footprint in the food industry is no longer merely an environmental indicator that needs to be reported; it has become a fundamental engineering criterion that guides process design. Carbon-negative approaches offer an innovative and strategic transformation area that enables the repositioning of the food engineering discipline within the sustainability axis.

Engineering Strategies in Zero-Waste Process Design

Achieving zero-waste goals in the food industry requires redesigning processes at the system level rather than relying on individual technological solutions. In this context, engineering strategies encompass a multidimensional approach ranging from raw material usage to energy and water management, by-product evaluation, and process integration (Ghisellini et al., 2016). Zero-waste process design should be approached as a holistic engineering problem that aims to simultaneously optimize production efficiency and environmental sustainability.

Process Integration and System-Level Optimization

Process integration refers to the joint evaluation of all unit operations in a production facility, considering energy and material flows. In the food industry, the traditional design of processes as independent units leads to energy losses and waste generation. However, system-level integration can significantly reduce waste by ensuring that heat, mass, and energy flows are compatible with each other (Klemeš et al., 2013).

Pinch analysis and similar process integration tools are used effectively in food plants, especially for heat recovery. Reusing waste heat from energy-intensive processes such as drying, pasteurization, and evaporation for purposes such as preheating or process water heating reduces both energy consumption and indirect carbon emissions (Smith, 2005). Such integration approaches are one of the fundamental engineering components of zero-waste process design.

Water Management and Closed-Loop Systems

The food industry is one of the industries that use large amounts of process water, and water consumption poses a significant environmental pressure. In zero-waste process design, water must be considered not only as a consumed resource but also as a recoverable process input. Closed-loop water systems enable process water to be reused through appropriate treatment and filtration techniques (Vourch et al., 2008).

Membrane filtration systems, advanced oxidation processes and biological treatment technologies are among the main engineering solutions that support water recovery in food processes. These systems reduce the amount of wastewater, while also decreasing energy and chemical consumption associated with water use. The literature clearly states that integrated water management strategies play a critical role in achieving zero-waste goals (Bixio et al., 2008).

Conversion of by-products and waste into value-added products

One of the most important components of zero-waste process design is the conversion of by-products and waste fractions generated during food processing into high-value-added products. Fruit and vegetable pomace, grain bran, dairy industry by-products, and oilseed meal fractions can be converted into functional food ingredients, animal feed raw materials or bio-based materials through appropriate physical, chemical or biotechnological processes (Mirabella et al., 2014; Galanakis, 2020).

The effectiveness of these conversion processes depends on the correct determination of process conditions and integration strategies. Reintroducing by-products into the production process not only reduces waste, but also creates economic value and increases the resource efficiency of food factories. In this respect, by-product evaluation strategies support both the environmental and economic sustainability of zero-waste process design.

Energy Recovery and Waste Energy Utilization

Energy recovery is one of the key engineering strategies that is employed in zero-waste process design. Reevaluating waste heat, biogas and other energy flows generated in food processing plants reduces both fossil fuel consumption and greenhouse gas emissions. Biogas production from organic waste through anaerobic digestion is a widely used approach for both waste management and renewable energy production (Zhang et al., 2014).

The integration of biogas systems into food processes enables part of the energy demand to be met within the facility and reduces the waste disposal burden. Furthermore, cogeneration and trigeneration systems contribute to zero-waste and carbon-negative goals by increasing energy efficiency (Scarlat et al., 2018).

Industrial Symbiosis and Regional Integration

Industrial symbiosis is a regional engineering approach that involves evaluating by-products generated at one production facility as inputs for another facility or industry. Industrial symbiosis applications in the food industry offer effective solutions that increase resource efficiency, particularly in organized industrial zones and food production clusters (Chertow, 2000).

Thanks to this approach, organic waste generated by food businesses can be directed to energy production facilities, processed water can be used for agricultural irrigation, and by-products can be used in different industrial applications. Industrial symbiosis is considered a strategic tool that enables the transfer of zero-waste process design from the facility scale to the regional scale (Lombardi and Laybourn, 2012).

Technologies Enabling Carbon-Negative Food Production

Carbon-negative food production processes leverage advanced engineering techniques aimed at not only mitigating greenhouse gas emissions but also facilitating net carbon removal across the entire food supply chain. This objective necessitates a comprehensive design that incorporates renewable energy systems, biomass conversion technologies, carbon capture mechanisms, and energy integration solutions, rather than focusing on isolated technological applications. The amalgamation of these technologies within the food industry transcends mere carbon footprint reduction, thereby making carbon-negative production models more viable (Minx et al., 2018).

Renewable Energy Integration

Integrating renewable energy sources into food processing is a crucial strategy for decreasing emissions and shifting towards carbon-negative production models in the long term. A significant proportion of energy demand within food processing facilities is attributed to heat requirements, especially in the low-to-medium temperature range. Consequently, solar-based process heat applications have been extensively discussed in the literature, particularly for dairy and dairy product processing, addressing needs such as heating and pasteurization as well as process heat integration (Masera et al., 2023). Likewise, biomass-based systems, including biomass boilers, present viable alternatives to fossil fuels for industrial process heat demands. This approach is especially relevant in the food industry to satisfy steam and hot water requirements in a low-carbon manner (IRENA, 2015a). Moreover, research focused on the water–energy–food nexus from a "system-level integration" perspective indicates that the incorporation of renewable energy can enhance sustainability performance through indirect benefits such as improved efficiency and reduced losses across the supply chain (IRENA, 2015b). Energy integration not only minimizes direct emissions but also bolsters the economic resilience of sustainable food production systems by enhancing security of energy supply. The literature suggests that food production facilities supported by renewable energy can achieve long-term carbon-negative objectives (Panwar et al., 2011).

Production of Biogas and Bioenergy from Organic Waste

Organic waste and by-products from the food industry are recognised as a vital energy resource within carbon-negative strategies, which is why it is so important to reduce, reuse and recycle. The production of biogas through anaerobic digestion not only facilitates the disposal of these waste materials but also fosters the generation of renewable energy. Utilising biogas for electricity and heat production enables food facilities to satisfy a substantial portion of their energy requirements on-site (Khalid et al., 2011).

Digestate, the by-product of anaerobic digestion, can be effectively employed in agricultural applications when properly assessed, promoting a closed-loop system within the food-energy-agriculture cycle. In this context, biogas systems are regarded as fundamental components of carbon-negative food processes (Scarlat et al., 2018).

Biochar Production and Carbon Sequestration

Biochar production is a prominent carbon-negative technology due to its ability to sequester carbon over the long term. By converting food processing by-products and agricultural waste into biochar through pyrolysis, a significant amount of the carbon found in biomass can be preserved in a stable form. This process contributes to net negative emissions by enabling carbon to be stored in soils for hundreds of years (Woolf et al., 2010; Lehmann et al., 2011).

From the food industry's viewpoint, biochar technologies present a strategic solution that simultaneously tackles waste management and carbon reduction. Additionally, the energy derived from by-products like synthesis gas and bio-oil produced during pyrolysis enhances the overall energy efficiency of the process (Lehmann and Joseph, 2015).

Capture, Use, and Storage of Carbon (CCUS)

In the food business, carbon capture, utilization, and storage (CCUS) technologies are becoming increasingly important, especially when it comes to controlling emissions from fermentation and biomass-based energy production processes. The food, beverage and agricultural industries can recover and repurpose pure CO2 streams produced during fermentation processes. By keeping carbon from being released into the atmosphere, this strategy supports circular carbon management (IEA, 2020).

Among carbon-negative technologies, carbon capture systems linked with biomass energy (BECCS) are seen to have the greatest promise. Long-term, BECCS applications in the food industry are anticipated to be crucial in the design of carbon-negative food factories, despite their current limited use (Minx et al., 2018).

Integration of Renewable Energy

One of the key technical techniques for lowering carbon emissions and eventually switching to carbon-negative production models is the incorporation of renewable energy sources into food processes. Process heat requirements in the low and medium temperature range (usually <200 °C) account for a large share of the food industry's overall energy demand. Because of this, producing process heat from renewable resources is a viable and efficient solution for the food industry (FAO, 2014; IRENA, 2015b).

Solar thermal systems have been extensively explored in the literature as alternatives that lower the use of fossil fuels in preheating, equipment cleaning (CIP systems), hot water production, and some pasteurization applications, especially in the dairy industry. Reducing greenhouse gas emissions while preserving process dependability is made possible by the hybrid integration of these systems with conventional energy sources (Sharma et al., 2017; Singh et al., 2019).

On the other hand, organic byproducts produced in food processing facilities have a great deal of potential for use in biomass-based energy systems. Steam and hot water can be produced by burning agricultural leftovers and food industry byproducts in biomass boilers. This strategy reduces emissions associated with fossil fuels while also integrating waste management with energy generation (IRENA, 2015b; Scarlat et al., 2018).

In the context of the water-energy-food nexus, renewable energy integration improves system efficiency in addition to directly lowering carbon emissions. Evaluating food production systems in conjunction with energy sources fosters resource efficiency and offers a comprehensive view of sustainable process design. Thus, while designing zero-waste and carbon-negative food factories, renewable energy integration should be viewed as a supporting infrastructure component (FAO, 2014; Purwanto et al., 2021).

Applications of Industrial-Scale Carbon-Negative and Zero-Waste Processes

To achieve zero-waste and carbon-negative objectives on an industrial scale, these strategies must be backed by engineering solutions that are both conceptually sound and practical in actual production settings. Systems that address process integration, by-product evaluation, energy recovery, and renewable energy integration are both economically and environmentally feasible, as shown by applications in several food industry subsectors (Ghisellini et al., 2016; Galanakis, 2015).

Integrated Methods in the Dairy Industries

In addition to using a lot of energy and water, the dairy business generates a lot of byproducts. Whey is a key component of industrial zero-waste efforts. Whey has long been considered an environmental problem due to its high biological value proteins, lactose, and mineral components; however, today, converting this fraction into protein concentrates and isolates, lactose-based products, or value-added components obtained through fermentation has become a common industrial practice. This change boosts the dairy industry's economic efficiency and drastically lowers waste (Smithers, 2008).

Heat recovery and biogas integration are important engineering solutions in milk processing facilities from the standpoint of energy management. Reusing waste heat from procedures like pasteurization, sterilization, and evaporation lowers overall energy needs and boosts process effectiveness. Additionally, waste management and energy generation can be integrated by using anaerobic digestion to turn organic byproducts and wastewater from the dairy industry into biogas. These applications can drastically lower dairy processing facilities' net energy usage and, as a result, their carbon footprint (Mata-Állvarez et al., 2000; Scarlat et al., 2018).

Integrated Methods in the Fruit and Vegetable Processing Industries

Large amounts of by-products, including peel, pulp, seeds, and stems, are produced by the fruit and vegetable processing sector. When treated properly, these fractions have a great deal of potential for additional value because they frequently include high concentrations of dietary fiber, phenolic compounds, natural colors, and antioxidants. One of the key uses of the zero-waste method in industry is the utilization of these by-products as natural additives, functional food ingredients, or raw materials for animal feed (Mirabella et al., 2014; Galanakis, 2020).

Fruit and vegetable processing facilities need a lot of energy during operations including drying, concentration, and cooling. Applications for waste heat recovery and energy integration are therefore essential for lowering carbon emissions. Heat pumps, inter-process heat integration, and multi-stage drying systems stand out as engineering solutions that improve energy efficiency. These strategies help to reduce operating expenses and the impact on the environment (Garnett, 2011).

Integrated Methods in the Fermented Beverage and Beer Industries

Because of byproducts like spent grain and wastewater with high organic loads, the beer and fermented beverage industry offers a great deal of promise for zero-waste applications. Because of its high fiber and protein content, spent grain can be used as a raw material for food ingredients, animal feed, or biotechnological uses. Successful instances of combining waste management with energy production can be found in the industrial use of this by-product for the generation of biogas or as a raw material for bio-based goods (Mussatto et al., 2006).

Another significant application taken into consideration within carbon-negative techniques is the capture and reuse of CO_2 streams produced during fermentation operations. By keeping carbon from being discharged into the atmosphere, the recovery of relatively pure CO_2 produced during the production of beer and other alcoholic beverages specifically aids in closed-loop carbon management. These applications are one of the workable and expandable ways to lower carbon emissions in the food sector (IEA, 2020).

Integrated Approaches in the Sugar and Starch-Based Industries

Because sugar beet and starch-based industries generate a lot of biomass byproducts, they are among the sectors that can be used for carbon-negative and zero-waste applications. Pulp and molasses are examples of by-products that can be used as raw materials in biochemical processes, as animal feed, or to produce biogas. In addition to lowering waste, these applications enable the recovery of materials and energy.

The extensive use of cogeneration systems in these industries is notable in terms of energy management. The facilities' net carbon balance is improved and energy efficiency is increased when heat and electricity are produced simultaneously from the same fuel input. In the sugar and starch-based sectors, cogeneration applications combined with biomass-based energy systems are regarded as important engineering solutions that help achieve carbon-negative targets (Scarlat et al., 2018).

Engineering and System Challenges in Transitioning to Sustainable Food Processes

Although the transformation towards zero-waste and carbon-negative targets in the food industry is technically feasible and has been described in detail in the literature, its widespread adoption on an industrial scale brings with it numerous engineering and system challenges. These challenges are not limited to the development of new technologies or their integration into existing processes; they require the simultaneous consideration of multi-layered factors such as the traditional design approach of production facilities, investment and operating costs, infrastructure compatibility, regulatory and institutional frameworks, and the simultaneous management of different resource flows.

The fact that a significant portion of food processing facilities are designed based on linear production models makes it difficult to implement energy recovery, by-product evaluation, and closed-loop systems. Adapting the existing infrastructure to be compatible with sustainability-focused technologies often necessitates extensive process modifications and additional investment requirements.

Furthermore, critical criteria such as food safety, hygiene, and product quality pose significant constraints that limit engineering flexibility in sustainable process design. From an economic perspective, the feasibility of zero-waste and carbon-negative applications is closely linked to energy prices, the stability of by-product markets, incentive mechanisms, and economies of scale.

While many sustainable technologies offer long-term environmental and economic benefits, they are approached with caution in industrial decision-making processes due to high investment costs in the short and medium term.

This situation stands out as a significant barrier limiting the widespread adoption of technically feasible solutions in practice.

In this context, the transition to sustainable food processes necessitates the adoption of process integration, system-level optimization, and multidisciplinary engineering approaches rather than isolated solutions. Holistic design approaches that consider energy, water and material flows together are critical for achieving zero-waste and carbon-negative targets at an industrial scale. This approach ensures that food engineering is repositioned as a discipline that focuses not only on production efficiency but also on environmental sustainability (Garnett, 2011; Ghisellini et al., 2016).

Technical and process-related challenges

One of the fundamental technical challenges encountered in zero-waste and carbon-negative process design is that existing food processing facilities are largely designed according to conventional production paradigms, which is something that needs to be addressed. Many industrial food facilities are built on a linear production model, where raw material input ends with product output and by-products are mostly removed from the system as waste. Such facilities often lack the infrastructure necessary for by-product recovery, energy integration and closed-loop water or material systems. Consequently, integrating new sustainability-focused technologies into existing processes requires extensive process modifications and additional engineering investments (Klemeš et al., 2013). Furthermore, food processes inherently demand high precision in terms of product safety, quality consistency, and regulatory compliance. Any changes to energy and material flows can directly or indirectly affect the microbiological stability, sensory properties, and shelf life of the product. In applications such as heat recovery, process water reuse, and the reintegration of by-products into the production line, hygienic design principles, the risk of microbial cross-contamination and the sustainability of process control pose significant engineering constraints.

In heat recovery systems, for example, improperly designed temperature levels and contact surfaces can lead to undesirable microbial growth or product quality deterioration. Similarly, in water reuse applications, inadequate treatment or monitoring systems can pose serious risks to product safety, which is why it is so important to have the right systems in place. The integration of by-products into the process requires careful engineering assessment in terms of compositional variability and process stability. In this context, it is imperative to address the balance between sustainability goals and product safety and quality requirements, together with advanced process control.

Economic Feasibility and Scalability Issues

One of the fundamental challenges encountered during the transition to sustainable food processes is the high initial investment costs and the uncertainty of the payback periods for these investments. Renewable energy systems, advanced water and waste treatment technologies, and processes aimed at converting by-products into high-value-added products often require higher capital investment compared to traditional production systems. This poses a significant barrier to the adoption of sustainability-focused technologies, particularly for small and medium-sized food businesses with limited financial flexibility (IEA, 2020).

Economic feasibility is not limited to investment costs alone, but also encompasses uncertainties regarding the long-term operational performance of sustainable processes. Fluctuations in energy prices, the uncertainty of carbon pricing mechanisms, and the unstable structure of by-product markets can increase the economic risk of sustainability investments. Converting by-products into high value-added products does not always provide a predictable revenue stream, as it depends on market demand, product standardization, and supply chain integration.

The industrial-scale application of laboratory or pilot-scale sustainable processes often requires technical and economic adaptations when evaluated in terms of scalability. Increased process complexity can raise maintenance and operating costs and negatively impact the economic performance of the system, which is why it is important to keep an eye on these issues. In this context, the literature emphasizes that developing modular, flexible, and scalable engineering solutions is critical for the widespread adoption of zero-waste and carbon-negative applications. Modular systems reduce financial risk by allowing for phased investment and provide adaptability to different business scales (Poore and Nemecek, 2018).

Consequently, technical, and process-related challenges are among the key factors limiting the feasibility of zero-waste and carbon-negative approaches, which necessitates the development of systematic, risk-based, and engineering-focused solutions in the design of sustainable food processes.

System Integration and Multi-Source Management

Sustainable food processes require that energy, water and material flows be considered as complex systems that are not independent of each other, but interact with one another. In this context, the Water–Energy–Food Nexus approach provides a holistic framework for the design and evaluation of food production systems. The Nexus approach enables the assessment of sustainability objectives at the system level by making visible the indirect effects of decisions regarding the use of one resource on other resources. However, translating this holistic perspective into industrial applications brings with it various technical and organizational challenges (FAO, 2014; Purwanto et al., 2021).

One of the fundamental challenges to system integration is data scarcity and heterogeneity in data quality. Data on energy, water and material flows in industrial food facilities are often collected at different time scales, with varying measurement accuracies and through separate systems. This situation increases uncertainty in integrated analysis and optimization efforts and complicates the development of reliable decision support mechanisms. Furthermore, the complexity of interactions between processes makes it difficult to accurately model system behaviors.

Industrial-scale energy integration and optimizations of waste streams require the use of advanced process modelling, simulation, and multi-objective optimizations tools. Applications such as heat integration, waste heat recovery, water reuse, and the redirection of by-product flows can yield meaningful results when considered not just on an individual process basis, but across the entire facility and even at a regional scale. However, the failure to sufficiently adapt such integrated analysis tools to industrial applications, or their lack of user-friendliness, is a significant factor that increases uncertainty in the design and operation phases of sustainable systems (Klemeš et al., 2013).

In addition, system integration necessitates an interdisciplinary approach. The simultaneous involvement of different disciplines such as food engineering, energy systems, environmental engineering, and economics requires coordination both technically and organizationally. Interdisciplinary communication gaps and inconsistencies in decision-making processes can complicate the implementation of integrated sustainability solutions.

Consequently, system integration and multi-source management stand out as one of the most critical yet complex engineering areas in the transition to sustainable food processes. Overcoming these challenges will be possible through the establishment of reliable data infrastructures, the widespread adoption of advanced modelling and decision support tools, and the strengthening of interdisciplinary collaborations. This approach will ensure that zero-waste and carbon-negative targets become feasible not only at a theoretical level but also on an industrial scale.

Regulatory Framework and Institutional Barriers

In the food industry, regulatory frameworks are important mechanisms that can both support and constrain the transition to sustainable processes. For example, food safety and hygiene standards may require the implementation of new technologies in production processes, while also necessitating detailed compliance procedures for the industrial-scale use of these technologies. Today, in many countries, food facilities are required to comply with proactive risk analysis-based inspection and control systems, and these requirements can create additional costs and time burdens in the adoption of innovative processes.

On the other hand, regulatory standards also establish the infrastructure for sustainability-focused processes to protect consumer health and ensure reliable quality for international trade (ISO 22000). Applications for the re-evaluation of waste and the inclusion of by-products in the food or feed chain are also under intense regulatory scrutiny.

Waste management and hygiene regulations in force in the European Union and other regions set out detailed conditions for the reintegration of by-products into the production process, which can complicate the adaptation processes of innovative technologies while attempting to strike a balance between environmental sustainability and food safety (Okumuş, 2024).

In addition to the regulatory framework, corporate and organizational factors also emerge as significant barriers to sustainable transformation. Elements such as internal organizational structure, technical knowledge base, employee competence, and resistance to change are important dynamics that directly influence the adoption of new process designs. Production habits and decision-making mechanisms integrated into current operations can create reservations about adapting sustainable engineering solutions. Such organizational barriers require a holistic assessment that encompasses not only technical performance criteria but also areas such as application capacity and management flexibility in the food sector.

Future Perspective: Holistic and Intelligent Systems

The success of the transition to sustainable food processes in the future will depend on the effective use of holistic system design, digitalization, and intelligent decision support mechanisms rather than individual technological improvements. The increasing complexity of food production systems requires the simultaneous monitoring and optimization of energy, water and material flows, rendering traditional, static process design approaches inadequate. In this context, supporting process integration, energy recovery, and by-product evaluation strategies with digital tools and data-driven methods will significantly facilitate achieving zero-waste and carbon-negative targets by increasing resource efficiency (Klemeš et al., 2013).

Digital twins, sensor-based monitoring systems, and advanced process modelling tools enable real-time performance evaluation of food factories, allowing for the dynamic management of energy consumption, waste generation and carbon emissions. Such smart systems not only optimize existing processes but also reduce uncertainty in decision-making processes by enabling the prior assessment of possible scenarios. Thus, sustainability goals can be addressed simultaneously with operational efficiency and product quality.

In this context, sustainable food factories are now being redefined not merely as product-producing facilities, but as integrated systems that generate energy, manage waste, convert by-products into added value, and actively control carbon balance. This transformation necessitates a broader systems approach in the food industry, extending from facility to supply chain scale. The discipline of food engineering plays a key role in this process in terms of system design, process integration, and interdisciplinary coordination, ensuring that sustainability goals are transformed into engineering-based, applicable solutions (Poore and Nemecek, 2018).

CONCLUSION

In the food industry, zero-waste and carbon-negative process design is gaining increasing strategic importance in the context of ensuring environmental sustainability and combating climate change. Increasing natural resource constraints, energy costs and global pressures regarding greenhouse gas emissions render the evaluation of food production systems based solely on production efficiency insufficient, and it is necessary to consider these systems alongside their environmental impacts. In this context, zero-waste and carbon-negative approaches are emerging as key transformation tools supporting the shift from linear production models to circular and regenerative systems in the food industry.

It is evident that sustainability in food processes can be achieved not through individual technological solutions, but through process integration, system-level management of energy and material flows, directing by-products towards high value-added applications, and integrating renewable energy-based solutions into production systems. Industrial-scale applications demonstrate that these approaches are technically and economically feasible under appropriate design and operating conditions, showing that sustainability goals can be addressed alongside production performance.

However, transitioning to sustainable food processes presents significant challenges due to limitations in existing plant infrastructure, high investment costs, market and demand uncertainties, and constraints arising from regulatory frameworks. This situation necessitates that sustainability-focused engineering solutions be evaluated not only in terms of technical performance but also according to criteria such as scalability, flexibility, system compatibility, and long-term operational performance. Increasing applicability at an industrial scale requires that engineering design and economic analysis be considered together.

Looking ahead, the success of sustainable transformation in the food industry is directly linked to holistic system design, interdisciplinary integration, and the effective use of digital decision support tools. Digitalization, advanced process modelling, and data-driven optimization approaches will enable more effective management of energy, water, and material interactions, thereby supporting the achievement of zero-waste and carbon-negative targets. In this context, the discipline of food engineering plays a central role in the design and implementation of sustainable food systems, contributing to the alignment of environmental goals with industrial realities through engineering-based solutions.

Consequently, zero-waste and carbon-negative process design is considered not only an environmental necessity for the food industry but also a strategic approach to enhancing long-term competitiveness, system resilience, and resource security. In this context, engineering-based, integrated, and scalable solutions are expected to make significant contributions to the widespread adoption of sustainable food production systems, which is why they should be used as much as possible.

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CHAPTER 6

PROCESS AND COST OPTIMIZATION IN THE PRODUCTION OF FUNCTIONAL CHEMICAL MATERIALS

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INTRODUCTION

Functional chemical materials, unlike conventional chemical products, are defined not only by their basic chemical composition but also by the specific performance properties they deliver, and they constitute high value-added industrial inputs. These materials are designed to fulfill particular functions such as electrical or thermal conductivity, optical properties (color, gloss, opacity), catalytic activity, barrier performance, antimicrobial action, rheological regulation, or surface functionality. Therefore, the production of functional materials often requires high purity levels, controlled crystal structure or particle morphology, a narrow particle size distribution, and consistent surface characteristics. These requirements render the production processes of functional chemical materials complex both technically and economically.

In these product groups, the key factor determining competitiveness is not merely the chemical formulation developed or the synthesis method used. The primary determinant is the ability to implement an integrated process design that can reduce the total cost per unit product while maintaining product performance and quality consistency. Modern chemical engineering literature emphasizes that process design must focus not only on technical feasibility but also on economic sustainability (Towler & Sinnott, 2022). In this context, functional material production is regarded as a multidimensional problem domain that must be addressed within the triangle of “chemical design–process engineering–economic analysis.”

From an engineering perspective, the production costs of functional chemical materials are not limited solely to raw material prices. The main factors shaping process costs include reaction yield and selectivity; separation and purification burden; energy consumption; equipment scale and material selection; process safety and loss-prevention strategies; maintenance and repair requirements; unplanned downtime; and risks associated with quality control and off-spec production. All of these technical parameters are concretized economically within the distribution of capital expenditures (CAPEX) and operating expenditures (OPEX) (Peters et al., 2003; Towler & Sinnott, 2022).

In particular, the CAPEX burden in functional material production can be higher than in conventional chemical production plants. The main reasons include the need for higher-grade equipment due to corrosion or solvent compatibility; special design requirements in terms of dust explosion and process safety; lines requiring high purity or contamination control; and sensitive drying–milling systems. In contrast, OPEX items are shaped by factors such as energy, auxiliary chemicals, solvent recovery, labor, maintenance, and waste management. This indicates that process design decisions in functional material production directly affect the long-term cost structure.

Another critical dimension of economic performance in functional materials is related to economies of scale. A product that exhibits high performance at laboratory or pilot scale may fail to deliver the expected cost advantage when produced at industrial scale due to changes in process dynamics. Factors such as mixing regimes, heat and mass transfer limitations, crystal growth kinetics, and particle agglomeration can lead to deterioration in product quality or increased energy consumption during scale-up. This reveals that scale-up in functional material production is not merely a capacity increase but also a quality and cost optimization problem (Peters et al., 2003).

At this point, energy efficiency and process optimization stand out as levers that both reduce costs and enhance competitiveness at industrial scale. Industrial energy consumption is concentrated particularly in unit operations such as drying, solvent recovery, high-temperature reactions, and milling. International Energy Agency (IEA) reports emphasize that energy efficiency investments not only reduce energy costs but also increase firms' resilience to market fluctuations and strengthen competitiveness (IEA, 2019; IEA, 2025). Within this framework, energy efficiency has become a strategic decision area in the production of functional chemical materials.

When considered together with energy efficiency, process optimization enables the restructuring of the cost profile in functional material production. Improving flowsheet design, reducing separation burden, increasing solvent and catalyst recovery, heat integration, and process intensification approaches can yield meaningful reductions in both CAPEX and OPEX. Towler and Sinnott (2022) emphasize that optimizations made at the early stages of process design provide significant economic gains throughout the entire life cycle of a plant.

From an economics perspective, functional chemical materials operate in competitive markets where cost increases cannot be reflected one-to-one in product prices. Therefore, manufacturers must treat cost control not as short-term expense reduction but as part of a long-term competitive strategy. Fluctuations in energy prices, raw material supply risks, and additional costs imposed by environmental regulations increase economic uncertainty in functional material production. In this context, process and cost optimization are considered key tools that enhance firms' ability to adapt to market conditions (IEA, 2019).

In recent years, the prominence of concepts such as sustainability, carbon footprint, and resource efficiency in industrial policies has made the process–cost equation even more critical in functional material production. Developing processes that consume less energy, generate less waste, and operate more efficiently is seen not only as an environmental requirement but also as an economic necessity. Industrial analyses show that companies investing in sustainable production technologies gain a stronger position in the long term in terms of cost advantage and market access (IEA, 2025).

Within the scope of this chapter, the interaction between process design and cost structure in the production of functional chemical materials is addressed at the intersection of engineering and economic perspectives. Process optimization, energy efficiency, scale-up, and economic assessment approaches are discussed as fundamental elements that determine competitiveness in functional material production. This holistic approach forms the basis of a modern chemical manufacturing understanding that centers not only technical performance but also economic sustainability.

ENGINEERING FUNDAMENTALS OF PROCESS OPTIMIZATION

In the production of functional chemical materials, process optimization plays a central role in establishing the balance between product performance and economic sustainability. The high value-added nature of such materials causes even small changes in process parameters to create pronounced effects on product quality, energy consumption, and unit cost. For this reason, process optimization should be treated as a holistic engineering approach encompassing not only improved operational efficiency but also quality consistency, process safety, and long-term cost control (Towler & Sinnott, 2022).

The complexity of optimization in functional material production requires the simultaneous evaluation of numerous unit operations and their interactions. Steps such as reaction, separation, drying, milling, and surface modification each rely on different physical and chemical principles and create different cost structures. In this context, process optimization necessitates a systematic consideration of the entire production line rather than the improvement of a single unit operation.

Definition of the Optimization Problem

Optimization problems in functional material production generally have a multi-objective structure. Manufacturers seek to achieve multiple targets simultaneously. These targets include cost minimization (€/kg or TL/kg), maximization of product performance (e.g., catalytic activity, mechanical strength, color strength, or functional surface area), reduction of environmental impact (energy consumption, emissions, and waste load), and the assurance of quality consistency and process safety.

It is often not possible to optimize all of these targets at the same time, and unavoidable trade-offs emerge. For example, achieving higher product purity or a narrower particle size distribution may require additional separation or classification steps, which can increase energy consumption and operating expenses. Similarly, process conditions selected to reduce energy consumption—such as lower operating temperatures—may negatively affect reaction rate or product performance. Therefore, the optimization process becomes a problem of balancing different objectives rather than maximizing a single one (Towler & Sinnott, 2022).

From an engineering viewpoint, optimization requires the mathematical and experimental definition of the relationships between decision variables and constraints. Decision variables refer to production conditions such as temperature, pressure, residence time, agitation speed, solvent ratio, solid/liquid ratio, recycle ratios, and equipment capacity parameters. Constraints consist of limiting factors such as product specifications, equipment limits, process safety requirements, and environmental regulations. The optimization problem aims to identify the most suitable solution by establishing a systematic balance among these variables and constraints (Peters et al., 2003; Towler & Sinnott, 2022).

From an economic perspective, this multi-objective structure directly affects manufacturers' decision-making processes. The optimal solution is often not the theoretically lowest-cost scenario, but rather an "economic optimum" at which technical risks, quality fluctuations, and market uncertainties are at acceptable levels. This indicates that process optimization must be based not only on engineering calculations but also on economic evaluations.

Flowsheet Development and Bottleneck Analysis

One of the first and most critical steps of process optimization is the accurate configuration of the production line flowsheet. Flowsheet design includes sequencing all unit operations from raw material reception to final product packaging, defining their interconnections, and determining material and energy flows. In functional materials, a typical flowsheet may begin with a reaction or synthesis step, followed by precipitation or crystallization, filtration or centrifugation, washing, drying, milling or classification, surface modification or coating, and finally packaging.

In such production lines, the cost burden is largely concentrated in separation, drying, and energy-intensive operations. While the reaction step often determines product value, separation and drying steps constitute a significant portion of total operating costs. Peters and co-authors (2003) report that separation and purification operations in chemical production plants can account for as much as 40–70% of total energy consumption. Therefore, eliminating unnecessary separation steps or replacing them with more efficient alternatives in flowsheet design is of great importance for cost optimization.

Bottleneck analysis is a complementary element of flowsheet optimization. A bottleneck is defined as the unit operation that limits capacity in the production line and determines the throughput of the entire system. Typical bottlenecks in functional material production arise in equipment such as drying capacity, filter area, centrifuge cycle time, or solvent recovery units. Correct identification of such bottlenecks is critical for the success of scale-up and capacity increase strategies (Peters et al., 2003).

Eliminating bottlenecks does not always mean investing in new equipment. Capacity increases can also be achieved through operational improvements such as rearranging process conditions, shortening batch times, increasing equipment utilization, or optimizing recycle streams. Such solutions limit CAPEX requirements while creating positive effects on OPEX.

Process Intensification

Process intensification stands out as one of the advanced engineering approaches to optimization in functional material production. Stankiewicz and Moulijn (2000) define process intensification as the development of safer, more efficient, and more compact production systems through the integration of unit operations, downsizing equipment, and enhancing heat and mass transfer.

This approach offers significant advantages, especially for high value-added functional materials. Micro- and mesoreactors, high-gravity equipment, integrated reaction–separation systems (e.g., reactive distillation), and membrane-based separation technologies are among the common application areas of process intensification. These technologies allow production with smaller reactor volumes by increasing reaction rates, while also reducing energy consumption and solvent use (Stankiewicz & Moulijn, 2000).

Baharudin et al. (2021) emphasize that process intensification approaches not only improve energy efficiency but can also yield significant reductions in CAPEX. Smaller equipment, less piping, and reduced utility requirements lower the overall investment cost of a plant. Moreover, compact plant designs offer advantages in terms of process safety and ease of maintenance.

Another important dimension of process intensification for functional materials is its impact on product quality. More controlled heat and mass transfer enables finer adjustment of particle size and morphology. This improves performance consistency, particularly for nanostructured materials or those for which surface functionality is critical. Thus, process intensification is regarded not only as a cost-reduction tool but also as a strategic approach supporting quality engineering.

COST STRUCTURE: CAPEX, OPEX, AND UNIT PRODUCT COST

In the production of functional chemical materials, the cost structure is a key indicator that directly reflects the economic outcomes of process design decisions. Such materials generally involve more complex production processes than conventional chemical products due to high performance expectations and narrow tolerance ranges. Therefore, cost analysis in functional material production requires not only the calculation of total expenditures, but also a holistic evaluation of the interaction between capital expenditures (CAPEX) and operating expenditures (OPEX) and how this interaction is reflected in unit product cost (Towler & Sinnott, 2022).

In chemical engineering literature, plant design and economic evaluation are regarded as activities that must be initiated at the early stages of a production process. Decisions made in the initial phases of process design—equipment selection, capacity determination, and flowsheet choices—define CAPEX and OPEX distribution throughout the life cycle of the plant. For this reason, the cost structure in functional material production should be treated as an integral part of technical feasibility (Peters et al., 2003).

CAPEX Components

Capital expenditure (CAPEX) covers all investments required to establish and commission a production facility. These expenditures include process equipment (reactors, separation units, dryers, milling and classification equipment), installation and assembly costs, piping systems, automation and instrumentation, electrical infrastructure, buildings and civil works, as well as commissioning and testing activities. CAPEX estimation and economic evaluation methods in the chemical industry are discussed in detail in foundational references such as Peters et al. (2003) and Towler and Sinnott (2022).

In functional chemical material production, factors that increase CAPEX largely stem from the quality and safety standards required by the product. In processes involving corrosion or aggressive solvents, the use of stainless steel (e.g., AISI 316L) or special alloys instead of carbon steel becomes mandatory, significantly increasing equipment costs. Likewise, in the production of functional materials in powder form, ATEX-compliant equipment and plant designs are required due to explosion risks, leading to additional investment costs.

In functional materials that require high purity, contamination control also plays a decisive role in CAPEX. Closed systems, lines operating under inert atmospheres, and specialized filtration and cleaning systems increase both equipment costs and plant complexity. Moreover, precision drying and particle size control equipment are indispensable in functional material production because they directly affect product performance. However, the high investment costs of these systems constitute a major share of the CAPEX budget (Towler & Sinnott, 2022).

From an economic standpoint, CAPEX decisions must be evaluated not only in terms of upfront investment costs but also together with payback period, financing costs, and investment risk. Especially for functional materials with high added value but limited market volume, the risk of overinvestment must be managed carefully. Therefore, modular plant designs and approaches that enable gradual capacity expansion from pilot scale stand out as strategies that reduce CAPEX risk (Peters et al., 2003).

OPEX Components

Operating expenditure (OPEX) covers the regular costs incurred throughout the period a plant is in operation. These costs include raw materials and auxiliary chemicals, energy (electricity, steam, cooling), labor, maintenance and repair, wastewater treatment, logistics, quality control, and general operating expenses. In functional chemical material production, the structure of OPEX varies significantly depending on the chemical nature of the product and process complexity.

For many functional materials, the largest components of OPEX are raw material and energy costs. In particular, raw materials of high purity or with specialized functional properties can constitute a substantial portion of total operating expenses. In addition, energy consumption is directly linked to reaction conditions and the intensity of separation and drying steps. Industrial analyses indicate that energy efficiency and process optimization are among the most effective levers acting on OPEX (IEA, 2025).

Another significant factor affecting OPEX in functional materials is maintenance and unplanned downtime costs. In production lines that use complex and sensitive equipment, maintenance requirements can be higher, and equipment failures can result in production losses. This increases not only direct maintenance costs but also indirect costs arising from lost production. Therefore, OPEX optimization must include not only the reduction of consumption items but also ensuring equipment reliability and process continuity.

From an economic perspective, OPEX is a cost component that is more sensitive to market conditions. Fluctuations in energy prices, disruptions in raw material supply chains, and environmental regulations can directly affect operating expenditures. Accordingly, functional material manufacturers treat OPEX management as a strategic decision area and invest in energy efficiency and process optimization to secure long-term competitive advantage (IEA, 2025).

ENERGY EFFICIENCY AND HEAT INTEGRATION: FROM ENGINEERING TO ECONOMICS

In chemical production processes, energy consumption is considered not only as an indicator of environmental sustainability but also as a strategic element that directly affects firms' cost structures and competitiveness. Especially under today's conditions—where energy prices fluctuate globally and geopolitical risks and supply uncertainties are increasing—energy efficiency has become a critical resilience parameter for production facilities. The International Energy Agency (IEA) identifies process optimization and energy management in industry among the main intervention areas to accelerate progress in energy efficiency (IEA, 2025).

The production of functional chemical materials is often energy-intensive due to high performance requirements. Drying, solvent recovery, high-temperature reactions, and mechanical operations carried out to preserve product purity, morphology, and functional properties constitute a major share of total energy consumption. This makes energy management in functional material production not merely an operational issue, but one of the key determinants of economic performance.

Energy-Intensive Unit Operations in Functional Material Production

In functional materials, energy consumption tends to be concentrated in specific unit operations. Drying processes, particularly when performed using equipment such as spray dryers, vacuum ovens, and fluidized-bed dryers, require large amounts of thermal energy. Because drying conditions influence not only energy consumption but also the particle size, surface area, and morphology of the product, optimizations in this step are critical both technically and economically.

Solvent recovery is another major source of energy consumption in functional material production. Distillation columns require substantial heat input, especially in high-boiling or multi-component solvent systems. At the same time, solvent recovery is often indispensable because it reduces raw material costs. This creates a complex optimization problem between energy consumption and raw material costs.

High-temperature reactions and thermal activation steps are other energy-intensive processes commonly used in functional material production. Although these steps are preferred to enhance product performance, they require careful engineering design in terms of energy consumption and equipment durability. Similarly, mechanical operations such as milling and classification can account for a significant portion of electricity consumption, especially when fine particle sizes are targeted.

Energy Efficiency: A Core Component of Process Optimization

Energy efficiency is recognized as one of the most effective levers for cost reduction in functional material production. Within process optimization, improving energy efficiency aims to achieve the same or higher production output with lower energy input. This approach reduces operating expenses (OPEX) and decreases firms' vulnerability to fluctuations in energy prices (IEA, 2025).

From an engineering standpoint, energy efficiency can be achieved through redesigning unit operations, optimizing process conditions, and minimizing energy losses. For instance, optimizing inlet–outlet air temperatures in drying, using moisture recovery systems, or evaluating alternative drying technologies can deliver significant energy savings. Likewise, optimizing the number of columns, tray/packing structures, and reflux ratios in distillation systems can markedly reduce energy consumption (Towler & Sinnott, 2022).

From an economic standpoint, investments in energy efficiency often have short to medium payback periods. This makes energy efficiency not only an environmental responsibility but also a financially rational investment area. IEA reports indicate that a large share of energy efficiency improvements in industrial facilities have payback periods of 2–5 years (IEA, 2025).

Heat Integration and Heat Recovery

Heat integration is one of the most fundamental engineering tools used to increase energy efficiency. It is based on reusing waste heat generated in one part of the process in other unit operations. Heat integration and heat recovery applications offer significant savings potential, especially in functional material production lines with multiple heating and cooling steps.

A commonly used method within heat integration is the design of heat exchanger networks where hot and cold streams are systematically matched.

Such networks reduce the need for external heating and cooling, thereby lowering total energy consumption. Towler and Sinnott (2022) state that a properly designed heat integration system can reduce the total energy requirement of a process by 10–40%.

The applicability of heat integration in functional material production depends on the thermal sensitivity of the product. For temperature-sensitive materials, heat integration must be designed carefully so as not to negatively affect product quality. Nevertheless, even utilizing waste heat at low temperature levels can provide meaningful energy savings in many cases.

The economic impact of heat recovery systems is observed directly on OPEX. Reduced energy consumption lowers operating expenses and shortens the investment payback period. Therefore, heat integration is regarded not only as a technical optimization tool but also as a strategic engineering decision that improves economic performance (Towler & Sinnott, 2022).

Economic and Competitive Effects of Energy Efficiency

The economic effects of energy efficiency and heat integration are not limited only to direct cost savings. Lower energy consumption increases the predictability of production costs and reduces the impact of energy price volatility on operations. This creates competitive advantage, particularly for functional material producers with high energy intensity.

Economic literature shows that firms improving energy efficiency tend to have more stable profit margins in the long term and demonstrate greater resilience to market shocks. In addition, energy efficiency investments contribute to lower carbon emissions, facilitating compliance with environmental regulations and strengthening firms' sustainability performance (IEA, 2025).

In functional material markets, effective management of the cost structure often means establishing a performance–cost balance rather than competing solely on price. Energy efficiency and heat integration provide critical tools for achieving this balance, enabling high-performance products to be produced at competitive costs.

SCALE-UP AND QUALITY CONSISTENCY: “FROM LABORATORY TO INDUSTRY”

In the development of functional chemical materials, the high performance values obtained at laboratory scale often cannot be preserved identically at industrial scale. The main reason is that scale-up is not merely an increase in reactor volume or production capacity; it also causes fundamental changes in flow, heat, and mass transfer regimes. These changes directly affect the microstructural properties of functional materials and thus the final product performance. Therefore, scale-up should be considered not only as a capacity increase problem but also as a quality engineering problem in functional material production (Peters et al., 2003; Towler & Sinnott, 2022).

Because functional materials often require a specific particle size distribution, crystal morphology, or surface functionality, even small deviations in process conditions can cause significant changes in product performance. Parameters that can be easily controlled at laboratory scale become more complex at industrial scale, introducing new sources of uncertainty. This makes it necessary to address the scale-up process through a systematic and staged approach.

Effects of Mixing and Mass Transfer

One of the most common issues in scale-up is changes in the mixing regime. While magnetic stirrers or small-volume mechanical agitators are used at laboratory scale, much larger agitators and different mixing geometries are employed at industrial scale. This change can reduce homogeneity in the liquid phase, disrupt the stability of solid–liquid suspensions, or create concentration gradients in the reaction medium.

Mass transfer limitations are particularly critical in functional material production processes involving precipitation, crystallization, or heterogeneous reactions. Mass transfer resistances that can be easily overcome at laboratory scale through high agitation rates may become pronounced in large-volume reactors. This can affect reaction rates and product formation kinetics, undermining quality consistency. Peters et al. (2003) emphasize that careful definition of mixing and mass transfer criteria during scale-up is essential for preserving product quality.

Heat Transfer and Thermal Effects

Another important dimension of scale-up in functional material production is heat transfer limitations. At laboratory scale, rapid and homogeneous heat transfer can be achieved thanks to a high surface area-to-volume ratio, whereas this ratio decreases significantly at industrial scale. This can lead to temperature gradients within the reactor and the emergence of localized overheating or overcooling zones.

Such changes in heat transfer have critical consequences, particularly for temperature-sensitive reactions or crystallization processes. Even small deviations in reaction temperature can influence crystal structure, purity, and functional performance. Towler and Sinnott (2022) note that insufficient consideration of heat transfer issues during scale-up can pose serious risks for process safety and product quality.

Crystallization Kinetics and Particle Size Distribution

A significant portion of functional materials is produced in crystalline or particulate form. In such products, crystal growth and nucleation kinetics are key factors determining final properties. While crystallization conditions are relatively homogeneous and controllable at laboratory scale, these kinetics can change at industrial scale due to flow and temperature gradients.

A shift in crystal size distribution during scale-up directly affects functional performance. For example, in materials such as pigments or catalysts, changes in particle size can significantly influence performance parameters such as color strength, surface area, or activity. Therefore, crystallization conditions must be carefully monitored during scale-up and re-optimized if necessary.

Effects of Drying Conditions on Morphology

Drying is a critical unit operation in functional material production with respect to both energy consumption and product morphology. Simple drying methods used at laboratory scale are replaced at industrial scale by more complex systems such as spray dryers, vacuum dryers, or fluidized-bed dryers. This change directly influences drying rate, moisture removal mechanisms, and particle structure.

Differences in drying conditions play a decisive role in particle agglomeration, porosity, and surface properties. This can affect functional material performance and shelf life. Therefore, in the scale-up process, the drying step must be handled carefully not only in terms of energy efficiency but also for preserving product quality.

Pilot Studies and Uncertainty Management

Pilot-scale studies are highly important for successful scale-up in functional material production. Pilot plants serve as a bridge between laboratory and industrial production and enable early identification of problems that may arise during scale-up. Through such studies, critical parameters-mixing, heat and mass transfer, crystallization, and drying—can be tested under conditions closer to industrial operation (Peters et al., 2003).

Uncertainty management is another key dimension of the scale-up process. Uncertainties emerging during scale-up create both technical and economic risks. Therefore, design and economics literature recommends supporting scale-up decisions with scenario analyses, sensitivity studies, and staged capacity expansion approaches (Towler & Sinnott, 2022). These approaches contribute both to reducing investment risk and ensuring quality consistency.

OPTIMIZATION STRATEGIES

Optimization strategies in the production of functional chemical materials aim not only to improve the process technically but also to secure economic sustainability and quality consistency. In this context, optimization is a multidimensional engineering problem that requires a holistic consideration of reaction conditions, separation–purification steps, drying and particle engineering, and process control and automation rather than improving a single variable. In the literature, this approach is referred to as “integrated process optimization” and is regarded as the key to competitiveness, especially in high value-added functional materials (Towler & Sinnott, 2022).

Reaction and Yield Optimization

The reaction step is one of the most decisive stages in functional material production in terms of both cost and quality. Reaction yield and selectivity directly affect raw material utilization, by-product formation, and thus the separation–purification burden. Improving selectivity not only increases the amount of target product but also reduces unwanted by-products, significantly lowering the cost of downstream processes.

Optimizing reaction conditions (temperature, pressure, residence time, reactant ratios) also plays a critical role in reducing energy consumption. Particularly for reactions requiring high temperature or pressure, even small improvements can result in meaningful reductions in operating costs. Towler and Sinnott (2022) emphasize that reaction optimization often affects OPEX rather than CAPEX and offers short payback periods.

In addition, catalyst and solvent recovery are important optimization strategies that reduce OPEX in functional material production. Reuse of catalysts or increasing solvent recovery rates reduces raw material costs and lowers environmental burden. Such strategies provide significant advantages in unit product cost, especially in continuous production lines.

Reducing the Separation–Purification Burden

In chemical processes, separation and purification steps typically stand out as the unit operations with the highest energy consumption and cost burden. Operations such as distillation, filtration, centrifugation, and membrane separation are indispensable in functional material production for ensuring product purity, yet they can constitute a major share of total OPEX.

Therefore, reducing the separation–purification burden is one of the core goals of process optimization. The process intensification approach aims to integrate reaction and separation steps or perform them with more efficient equipment. Reactive distillation, membrane-assisted reaction systems, or in situ product removal strategies are among the prominent solutions in this context (Stankiewicz & Moulijn, 2000).

Such integrated approaches can reduce CAPEX by decreasing the number of equipment units and positively affect OPEX by limiting energy consumption. Given the high purity requirements of functional materials, considering separation strategies together with reaction design is regarded as a critical optimization approach from an economic standpoint.

Drying and Particle Engineering

The drying step plays a decisive role in functional material production in terms of both energy consumption and product quality. Different technologies such as spray dryers, vacuum dryers, or fluidized-bed dryers have different impacts on product morphology. The drying profile directly influences critical properties such as particle size, porosity, surface area, and agglomeration tendency.

Therefore, drying optimization should be addressed not only from an energy efficiency perspective but also from a product performance perspective. Rapid drying at high temperature may increase energy consumption and cause undesirable changes in product morphology. In contrast, more controlled and lower-temperature drying strategies can improve product quality while optimizing energy costs.

The particle engineering approach aims to systematically analyze and control the impact of drying conditions on product performance. This approach is an important optimization tool that enables consistent attainment of targeted performance values at industrial scale in functional materials.

Process Control and Automation

Process control and automation systems are indispensable for the sustainable implementation of optimization strategies. Automation enables continuous monitoring of process variables (temperature, pressure, flow rate, pH, particle size, etc.) and rapid correction of deviations. This reduces off-spec production rates, thereby lowering both raw material losses and reprocessing costs.

Modern process design literature emphasizes that control strategies are an integral part of process optimization. Towler and Sinnott (2022) note that failing to consider controllability at the design stage can lead to unexpected cost increases and quality issues at industrial scale. In particular, advanced control strategies (model-based control, statistical process control) and automation-assisted monitoring systems increase capacity utilization and reduce unit product cost.

Another important contribution of process control in functional materials is ensuring quality consistency. Automation makes it possible to keep production conditions within narrow tolerance ranges and minimize variability in product properties. This increases customer satisfaction and contributes to brand value and economic sustainability in the long term.

CONCLUSION AND OVERALL ASSESSMENT

In the production of functional chemical materials, process and cost optimization presents a multidimensional structure that cannot be addressed merely as a technical efficiency problem within today’s industrial and academic discussions. Optimization efforts in this field are directly linked to strategic factors such as competitiveness, sustainability, supply chain resilience, and long-term economic stability. Therefore, functional material production necessitates an interdisciplinary approach in which chemical engineering principles are integrated with economic analyses.

The framework addressed in this book chapter demonstrates the importance of evaluating cost, quality, and environmental impact together from the early stages of process design in the production of functional chemical materials. As emphasized in the literature, flowsheet improvements made in the early phases of process design, reduction of separation burden, energy efficiency practices, and process intensification strategies can generate permanent and multiplicative impacts on both capital expenditures (CAPEX) and operating expenditures (OPEX) (Towler & Sinnott, 2022; Stankiewicz & Moulijn, 2000).

Relationship Between Process Design and Competitiveness

Functional materials are often high value-added products with sensitive technical properties and high market expectations. In these products, competitiveness depends not only on performance values achieved at laboratory scale but also on the ability to produce that performance consistently, predictably, and economically at industrial scale. Mixing, heat and mass transfer limitations, crystallization kinetics, and changes in drying conditions encountered during scale-up make it difficult to preserve product quality. Therefore, as highlighted in this study, scale-up should be treated as a holistic quality engineering problem rather than merely a capacity increase process.

Another fundamental dimension of competitiveness is the effective management of the cost structure. Balanced optimization of CAPEX and OPEX components increases functional material producers’ resilience to price volatility, rising energy costs, and supply chain vulnerabilities. In particular, optimizing energy-intensive unit operations (drying, distillation, milling) not only lowers unit product cost but also strengthens the firm’s financial flexibility.

Energy Efficiency and Economic Resilience

As discussed throughout this chapter, energy management is a core component not only of environmental sustainability but also of economic resilience. International Energy Agency (IEA) reports clearly demonstrate that energy efficiency investments in the industrial sector are strategic interventions that enhance competitiveness (IEA, 2019; IEA, 2025). In a global economic environment where energy costs are volatile, efficiency improvements for firms with energy-intensive processes are not merely cost advantages but strategies for survival.

In functional chemical material production, energy consumption is mostly concentrated in downstream processes such as separation and drying. Therefore, evaluating heat integration, waste heat recovery, and alternative low-temperature technologies reduces OPEX and shortens payback periods. From an economic perspective, such engineering solutions contribute to making capital investments more rational and sustainable.

Interdisciplinary Dimension of Optimization

One of the main outcomes of this book chapter is that treating process and cost optimization solely as an engineering problem is insufficient. Optimization decisions are directly related to economic factors such as investment risks, market uncertainties, regulatory pressures, and environmental costs. In this context, analyses that combine chemistry, engineering, and economics produce more realistic and applicable outcomes for both academic and industrial decision-makers.

Especially for niche, high value-added products such as functional materials, the impact of process design decisions on cost structure emerges in the long term. A process that is incorrectly or incompletely optimized may appear feasible in the short run, yet lead to outcomes such as high operating costs, quality problems, and loss of competitiveness in the long term. Therefore, optimization should be viewed as a holistic decision process in which not only technical parameters but also economic scenarios, sensitivity analyses, and risk assessments are considered together.

Sustainability and Future Perspective

In the production of functional chemical materials, sustainability has become a strategic necessity due to increasing regulations and societal expectations.

Approaches such as energy efficiency, waste minimization, solvent recovery, and process intensification reduce environmental impacts while directly contributing to cost optimization. This shows that sustainability is not merely an environmental policy but also a strong economic strategy.

Looking ahead, digitalization, data-driven optimization, process analytical technologies (PAT), and AI-assisted control systems are expected to become more widespread in functional material production. These technologies will enable more precise control of process variables, improved quality consistency, and early detection of cost deviations. Thus, process and cost optimization will evolve from a static design problem into a dynamic management process characterized by continuous improvement.

OVERALL CONCLUSION

In conclusion, the analyses presented in this book chapter reveal the multidimensional nature of process and cost optimization in the production of functional chemical materials. It is clearly seen that engineering decisions made in the early stages of process design are decisive for long-term economic performance. Energy management, process intensification, scale-up strategies, and automation practices stand out as fundamental tools that support both technical success and economic sustainability.

In this context, approaches integrating chemistry–engineering–economics enable the development of not only more efficient but also more resilient and competitive production models in functional material production. This holistic perspective contributes both to the academic literature and to the establishment of a guiding framework for industrial applications.

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CHAPTER 7

COMPARISON OF THE EFFECT OF SUBSTRATES IN TERMS OF HARDNESS/SCRATCH PROPERTIES OF CrN COATED

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INTRODUCTION

Surface modification techniques are widely employed to enhance the thermal, electrical and mechanical performance of materials, particularly properties such as hardness and resistance to wear. For this purpose, various surface engineering processes including welding, physical and chemical vapor deposition, thermal spraying and electrochemical treatments are applied. Among the characteristics of surface coatings, adhesion is considered the most critical parameter, regardless of the deposition technique utilized. To evaluate adhesion performance, several acceptable testing procedures have been proposed in the literature (Valli, 1986). These include the direct pull-off method (Elshabasy, 1981), accelerated force approaches such as ultracentrifuge (Campbell et al., 1970) and ultrasonic tests (Faure et al., 1972), acoustic imaging techniques (Bray et al., 1980), laser-based tests (Aindow et al., 1984), indentation methods (Mehrotra & Quinto, 1985; Batista et al., 2003; Fox-Rabinovich et al., 2004) as well as scratch testing procedures (Eryılmaz et al., 1997; Batista et al., 2002; Kamminga et al., 2004; Dobrzański et al., 2005; Dobrzański et al., 2008). These techniques are generally easy to apply, provide rapid results and show good repeatability. Several researchers have focused on the properties of CrN coatings. Bayón et al. (2009) analyzed multilayer Cr/CrN systems with varying layer thicknesses deposited on silicon and F1272 steel substrates, where wear and corrosion behaviors were examined together with hardness and compositional analyses. Fuentes et al. (2009) studied cathodic arc PVD-grown CrCN coatings with nitrogen–carbon concentration gradients to obtain low-friction surfaces and reported enhanced adhesion performance for CrN layers. Lamastra et al. (2006) employed X-ray diffraction (XRD) to determine residual stresses in CrN/Cr/CrN multilayer structures produced by cathodic arc evaporation on AISI H13, AISI 1040 and K340 substrates. Furthermore, Pengfei and Bailing (2011) explored the influence of carbon incorporation on the tribological behavior of CrN coatings deposited on single-crystal silicon and M2 high-speed steel (HSS). Their findings indicated that CrN layers possess a compact columnar morphology, with a friction coefficient close to 0.75 and a hardness value of approximately 1930 HV. Despite the large number of studies reporting CrN coatings on diverse substrates, it remains challenging to clearly evaluate the role of substrate type on the mechanical performance of these coatings, as the experiments have been conducted in different laboratories using varying surface preparation procedures.

Therefore, apart from prior works, in this study, CrN coating was deposited at the same laboratory and with same surface treatments on five different types of steels (DIN 1.7225, 1.2067, 1.2379, 1.2842 and K390) by cathodic arc deposition method. A comparative study was performed on the basis of scratch tests, dynamic micro-hardness tests, coating thickness measurement tests (Calotest) and Rockwell C adhesion tests.

EXPERIMENTAL STUDY

Substrate Preparation

Steel rods were obtained from Önerler Makina Çelik San. Tic. Ltd. Company. The substrates were prepared with the dimension of 100 mm length and 24 mm diameter. Chemical composition of the steel substrates is given in Table 1. There are many usages of these selected steel in the industry. These steels are used in production of parts in automobiles, construction and agriculture machines which require high quality, tool machines, airplane parts, axels, crankshafts, gears, bandages, bolts, nuts, journals, spiral drills, reamers and roller mill etc.

Table 1. Alloying elements in chemical compositions of steels (% weight)

Steels	C	Mn	Cr	Mo	V	Si	W	Co
DIN 1.7225 (AISI 4140)	0.41	0.83	0.9	0.18	---	0.21	---	---
DIN 1.2067 (AISI L3)	1.0	0.35	1.5	0.1	---	0.25	---	---
DIN 1.2842 (AISI O2)	0.90	2.00	0.40	---	0.10	0.30	---	---
DIN 1.2379 (AISI D2)	1.55	0.40	11.8	0.80	0.80	0.30	---	---
K390 BOHLER	2.47	0.4	4.2	3.8	9.0	0.55	1.0	2.0

All steel rods were first turned to a diameter of 13 mm and subsequently subjected to heat treatment, the details of which are summarized in Table 2. Following this process, the hardness of all specimens was determined to be 56 ± 1 HRc. After heat treatment, the diameters were further reduced to 12.7 mm using a fine-abrasive grinding wheel in order to remove any dimensional changes that may have occurred during thermal processing. To eliminate residual quenching oil from the substrate surfaces, the samples were immersed for a predetermined duration in a hot alkaline ultrasonic bath and then rinsed with isopropyl alcohol prior to the coating stage.

Table 2. Heat treatment conditions for steels

Steels	Quenching media	Quenching Temp (^{0}C)	Tempering Temp (^{0}C)
DIN 1,7225 (AISI 4140)	Oil	850	190-210
DIN 1,2067 (AISI L3)	Oil	920	200-220
DIN 1,2842 (AISI O2)	Oil	820	280-300
DIN 1,2379 (AISI D2)	Gas quenched in vacuum furnace	920	510-530
K390 BOHLER Micro Clean Steel	Gas quenched in vacuum furnace	1050	600-620

CrN PVD coatings were deposited on all of the steels using cathodic arc coating method. All of the steels were coated at a temperature in the range of 450-500°C. Fig 1 shows the schematic representation of coating process.

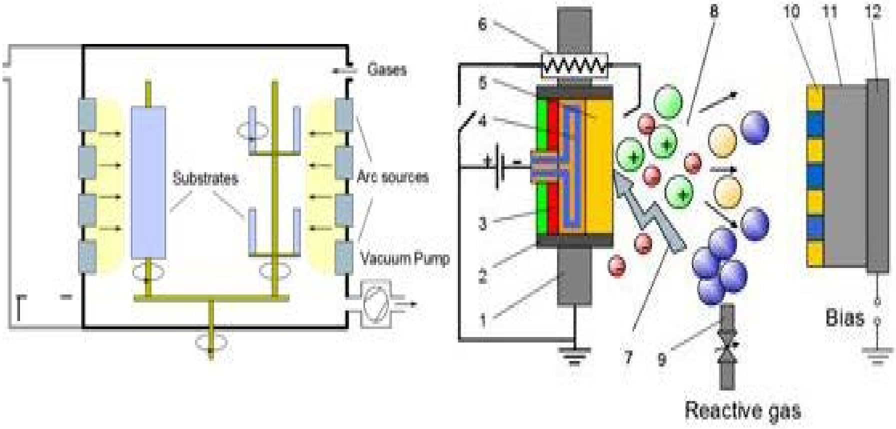

Figure 1: The Schematic representation of the coating process (1, Part of the coater wall, 2, Insulator, 3, Magnet system, 4, Cooled copper plate, 5, Target, 6, Ignitor, 7, Arc, 8, Ionized metal vapor, 9, Reactive gas inlet, 10, Coating, 11, Tool, 12, Tool holder)

Characterization Tests

All experimental evaluations were conducted after the deposition of the CrN layers in order to characterize the coated substrates. Initially, coating thickness measurements were performed by means of abrasion testing using a CSM Calotest system together with a 0.5 μm diamond slurry. Due to the cylindrical geometry of the samples, the abrasion process resulted in elliptical wear scars on the coated surfaces. The major and minor radii of these elliptical marks were subsequently determined with the aid of an optical microscope and dedicated image analysis software (Fig. 2).

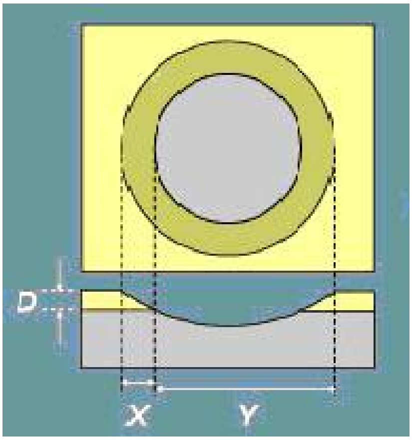

Figure 2: Schematic representation of the Calotest

Measured radius values were used in Eq, 1 to calculate the thickness of the coatings. This test also helped us to detect if the coating was a single layer and to check if there were any impurities within the coatings, coating thickness, t, is calculated following equation;

$$t = \frac{XxY}{\phi_{tip}} \tag{1}$$

Where t is the coating thickness. X and Y are the outer radius and inner radius, respectively. Øtip is the radius of the tip.

Scratch adhesion experiments were performed on all coated steel samples in accordance with ASTM C162 (2005) using a CSM Revetest system. For each substrate, five independent scratch tracks were generated. A Rockwell-type conical diamond indenter with a tip radius of 50 µm was employed under a progressively increasing normal load. The test parameters were set as follows: initial load of 5 mN, final load of 200 mN, loading rate of 136500 mN/min and a scratching speed of 7 mm/min. During the tests, critical load values corresponding to coating failure were continuously recorded through an acoustic emission sensor. In addition to acoustic signals, normal force, tangential force and the resulting friction coefficient were simultaneously monitored. The applied normal force induces coating damage; however, the load levels at which specific failure modes occur are defined as critical loads. In this study, three characteristic critical loads were identified in line with ASTM C162. The first, Lc1 (threshold load), corresponds to the onset of vertical cracking extending toward the substrate. The second, Lc2, represents the maximum load-bearing capacity, characterized by partial delamination of the coating along the crack edges. The third parameter, Lc3, denotes complete coating removal, indicating total detachment along the scratch track.

Microhardness measurements of the coated surfaces were conducted using a Fischer Dynamic Microhardness Tester (Fischer HM100) following ISO 14577-1 (2002). The experiments were carried out with a loading rate of 0.5 mN/s over a period of 20 s employing a standard Vickers indenter.

Furthermore, Rockwell C adhesion evaluations were applied to all specimens in accordance with DIN CEN/TS 1071-8 (2004). For this test, the substrate hardness must exceed 54 HRc to ensure reliable results, as the method is highly sensitive to both substrate hardness and coating thickness. As stated previously, all steels used in this work exhibited hardness values of 56 ± 1 HRc after heat treatment. This method does not provide a quantitative adhesion value; instead, adhesion quality is classified visually between HF1 and HF6 using standardized reference images. Ratings between HF1 and HF4 indicate sufficient adhesion performance, whereas classifications higher than HF4 reflect inadequate bonding strength. Following the Rockwell tests, surface damage was examined by optical microscopy at a magnification of 50X.

RESULT AND DISCUSSION

In order to characterize the coating, surface of the coating was examined by SEM-EDX. Fig. 3 shows the surface of the coating and EDX analysis. After

SEM examination, mechanical tests were performed. EDX results show that, for CrN coating, the C peaks come from main metal due to high voltage acceleration as 19 kV. CrN is in NaCl structure. Cr is located at the edges and center of the cube, while N is located at the corners and surface centers of the cube, or vice versa to provide CrN ratio as 1-1, respectively. When the molar ratios of elements were calculated by EDX wt%, the average CrN formation can be evaluated as Cr0.21N0.79. Since N ratio is less than desired, this composition can also be said metallic but harder and tougher than CrN due to matching coating elements with steel material.

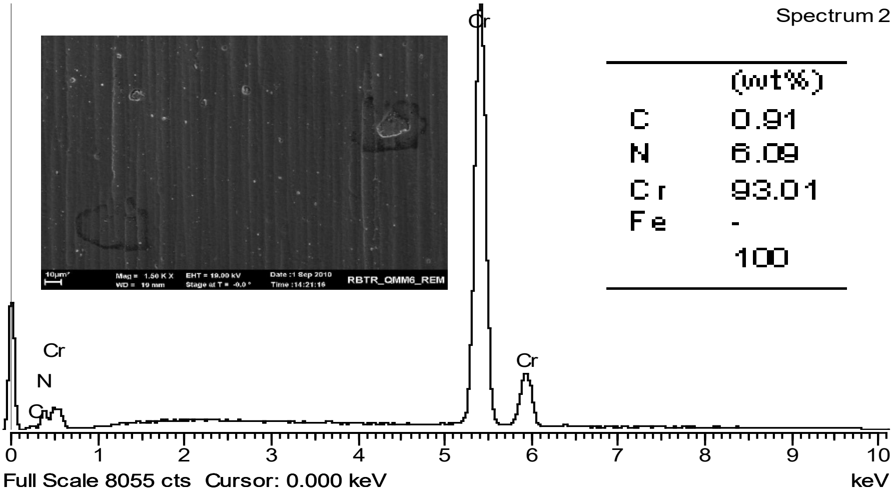

Figure 3: The surface of the coating and EDX analysis

Coating Thickness

CrN coating thickness was measured using a CSM Calotest Abrasion Instrument. Table 3 shows the test results. While the maximum average CrN coating thickness was obtained from DIN 1.2842 steel, the sequence of coating thickness is K390, DIN 1.2067, DIN 1.2379 and DIN 1.7225 steel substrates from higher to lower.

Table 3. Calotest thickness measurement test results

Steels	t_1,(µm)	t_2,(µm)	t_3,(µm)	t_{av},(µm)
DIN 1.7225	1.99	2.58	1.89	2.15
DIN 1.2067	2.98	2.98	1.84	2.57
DIN 1.2842	2.88	2.95	2.88	2.89
DIN 1.2379	2.16	2.37	2.95	2.49
K390	2.71	2.92	2.57	2.73

Scratch Test Results

The first critical load, Lc1, was extracted from the acoustic emission responses recorded during the scratch experiments. Nevertheless, the detection of such acoustic signals is strongly dependent on the thickness of the deposited coating. Valli et al. [1] employed scratch testing to investigate TiN layers on M2 high-speed steel and ASP30 sintered high-speed steel and concluded that a minimum coating thickness of 1.5 µm was required to reliably generate measurable acoustic emissions. In the present study, the CrN coatings exceeded this critical thickness, which enabled clear acquisition and evaluation of the acoustic emission data. The corresponding scratch test behavior of the DIN 1.2842 steel substrate is presented in Fig. 4.

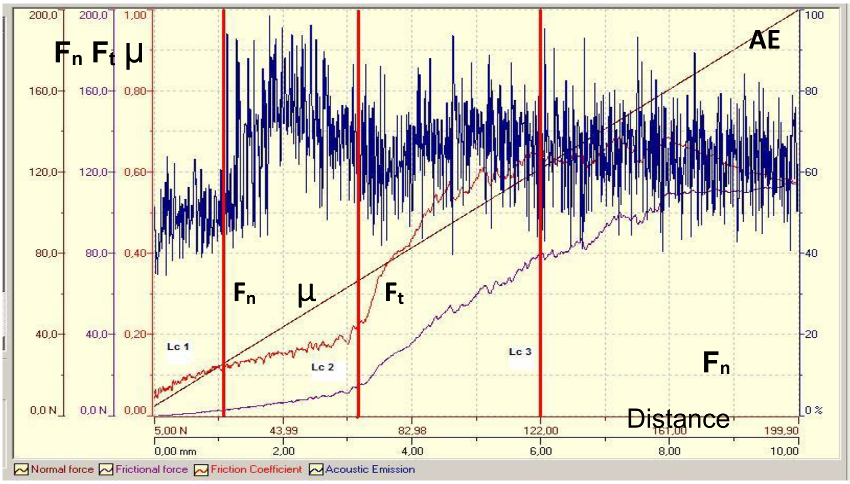

Figure 4: Scratch test of DIN 1,2842 steel

Evaluation of the experimental data indicates that three main variables-namely acoustic emission, friction force and friction coefficient-play a decisive role in identifying the critical load values. The first critical load, Lc1, was assigned to the point at which a sudden increase in the acoustic signal was observed. The second critical load, Lc2, corresponds to the region where both the friction force and the friction coefficient simultaneously rise with a noticeably steeper gradient. The third critical load, Lc3, was defined at the stage where the acoustic emission and friction coefficient curves begin to exhibit linear behavior. The scratch tracks were examined using an optical microscope at a magnification of 200×. Figure 5 presents typical damage features corresponding to micro-crack initiation (Lc1), the maximum load-bearing capacity (Lc2) and the total delamination of the coating (Lc3) for DIN 1.2067 steel.

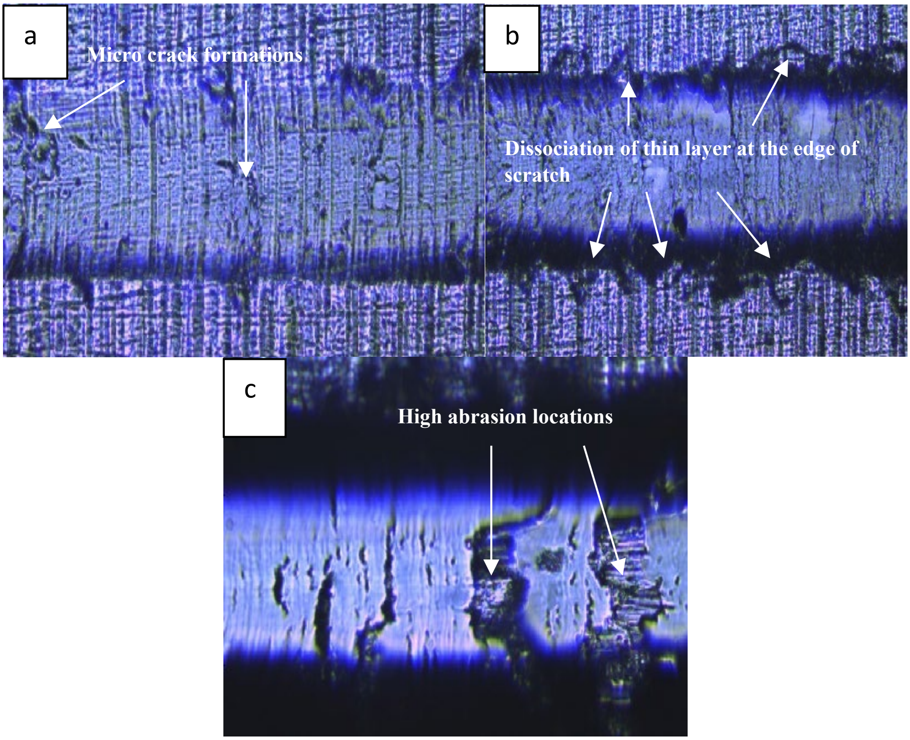

Figure 5: Microstructure at the critical loads obtained from scratch test for DIN 1,2067 steel a) Lc1; Micro-crack; b) Lc2; Dissociation of a coating layer at the edge of scratch; c) Lc3; Full separation of coating from steel

Results of the scratch tests were given in Table 4. Five tests were carried out for each substrate and their average scratch test results are listed in Table 4.

Table 4. Scratch test results of different steel substrates

CrN	Lc	F_n (N)	F_t (N)	μ	Distance (mm)
DIN 1.7225	Lc1	24.0	3.4	0.13	1.02
	Lc2	43.8	8.9	0.20	1.99
	Lc3	97.4	60.2	0.61	4.74
DIN 1.2067	Lc1	24.5	2.8	0.11	1.00
	Lc2	63.8	14.9	0.23	3.02
	Lc3	113.7	77.9	0.68	5.57
DIN 1.2842	Lc1	27.43	3.29	0.11	1.14
	Lc2	70.56	16.12	0.22	3.36
	Lc3	121.55	78.00	0.63	5.97
DIN 1.2379	Lc1	33.07	4.302	0.12	1.44
	Lc2	100.12	26.76	0.26	4.87
	Lc3	160.52	97.70	0.60	7.97
K390	Lc1	27.27	3.19	0.11	1.14
	Lc2	68.30	16.27	0.23	3.24
	Lc3	121.61	78.76	0.64	5.98

The CrN coating properties of steel substrates were compared on the basis of normal force (Fn). DIN 1.2379 steel have the highest mean Lc1, Lc2 and Lc3 critical load values among the other type of steel substrates, i,e. Loads required are the highest values for the crack formation (Lc1), load bearing capacity (Lc2) and removing of coating (Lc3). In addition to that, these highest load values were obtained in a longer distance. All these outcomes showed us that CrN coating adhered to DIN 1.2379 steel better than other type of steel substrates.

Friction coefficient, μ, values were also determined at Lc1, Lc2 and Lc3, DIN 1.2379 steel appeared to have almost the same friction coefficient values at mean Lc1. However, for mean Lc2 values, DIN 1.2379 steel have higher friction coefficient than the other type of steel substrate. The reason was considered to be the influence of normal force on friction coefficient. When the normal force on DIN 1.2379 steel increases, the friction coefficient and friction force (Ft) increase. It can be concluded that CrN coating on DIN 1.2379 gives better results than on the other type of steels.

Dynamic Micro-hardness Test Results

Dynamic microhardness measurements were conducted ten times on each substrate, after which the poorest result was discarded to enhance data reliability. Consequently, the remaining nine indentations were used for subsequent evaluations. Figure 6 illustrates the load–penetration depth curves obtained from these nine tests together with the software-generated average profile (shown in red) for the DIN 1.2842 steel sample.

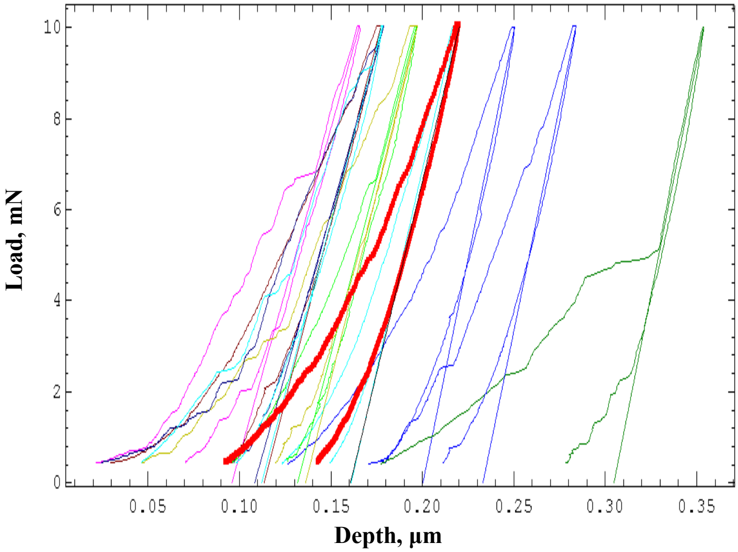

Figure 6. Load-Depth diagram obtained after dynamic micro-hardness test for DIN 1,2842 steel substrates. The graphs include 9 tests and the average curve (red curve)

Dynamic micro-hardness test results for both of the steels are listed in Table 5. As it is seen in the results in Table 5, micro-hardness (HV) values of CrN coating on DIN 1.2379 steel is greater than that of the other steels and elastic modulus (E) values of CrN coating on DIN 1.2067 and K390 steel are greater than that of the other type of steels.

Table 5. Dynamic micro-hardness test results

	Property, P	
	Hv	E, GPa
DIN 1,7225	2397	288
DIN 1,2067	2513	293
DIN 1,2842	2343	257
DIN 1,2379	2869	287
K390 BOHLER	2412	293

Rockwell C Adhesion Test

This is the most commonly used test in industry for determining the quality of a coating. Fig. 7 shows the Rockwell C adhesion test marks on the surface of steels respectively. As it is seen in Figure 7a. CrN coating was dissociated from the surfaces of DIN 1.2067, DIN 1.2482 and K390 steels by the applied force and the HF value was determined to be 4.5, 6 and 5 using the reference images for comparison by the software, respectively. This coating can be considered to be insufficient for usage since its HF value is higher than 4. On the other hand, Figure 6a and 6d show HF values of 1 for DIN 1.7225 and DIN 1.2379 steel substrates. This means the CrN coatings on DIN 1.2379 and DIN 1.7225 steel substrates have significantly better adhesion strength compare to the other steel substrates.

Rockwell C adhesion test results are listed in Table 7. As seen from Table 7, while, HF values of substrate DIN 1.7225 and DIN 1.2379 are 1 for three measurements, for K390 substrate it is 4 and above. Therefore, only substrate DIN 1.7225 and DIN 1.2379 have better adhesion in all substrates.

Table 7. Rockwell C adhesion test results

	DIN 1.7225			DIN 1.2067			DIN 1.2842			DIN 1.2379			K390		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
HF	1.0	1.0	1.0	1.0	4.0	4.5	1.0	5.0	6.0	1.0	1.0	1.0	5.0	4.5	4.0
Result	✓	✓	✓	✓	✓	X	✓	X	X	✓	✓	✓	X	X	✓

Table 8. Substrate Carbon Content and CrN Coating Hardness. Elastic Module and their relative values

Substrate Number	Substrates	% C	Hardness. Hv	Relative Hardness	Elastic Module. GPa	Relative Elastic Module
1	DIN 1.7225 (AISI 4140)	0.41	2397	84	288	98
2	DIN 1.2067 (AISI L3)	1.00	2513	88	293	100
3	DIN 1.2842 (AISI O2)	0.90	2343	82	257	88
4	DIN 1.2379 (AISI D2)	1.55	2869	100	287	98
5	K390 BUEHLER	2.47	2412	84	293	100

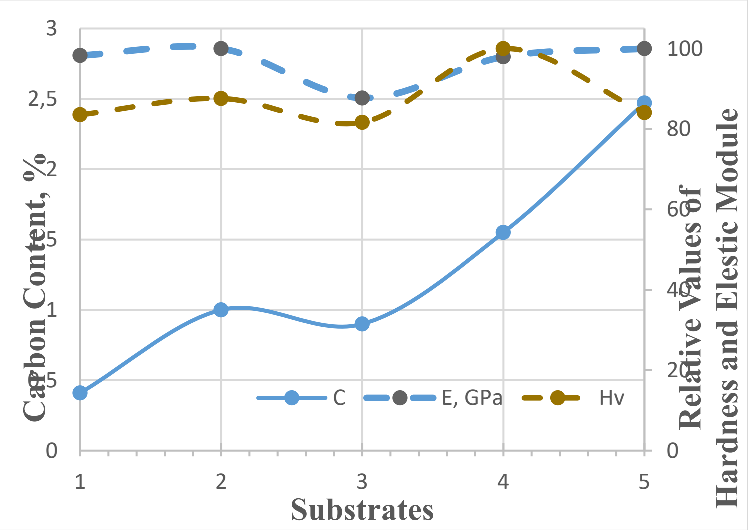

Figure 8: Substrate Carbon Content and CrN Coating Hardness and Elastic Module Relation

As can be observed from Table 8 and Figure 8. CrN coating hardness and elastic module change have same tendency as carbon content. It may be concluded that if the carbon content of the steel substrate increases the hardness and elastic module of the CrN coating. This may be deeply studied by checking bonding strength of CrN to carbon containing phases.

CONCLUSION

In this experimental study. CrN was coated on various steels called DIN 1.7225. DIN 1.2067. DIN 1.2842. 1.2379 and K390 by cathodic arc PVD method. The following conclusions are obtained;

1. The sequence of coating thickness is DIN 1.2842, K390, DIN 1.2067, DIN 1.2379 and DIN 1.7225 steel substrates from higher to lower.
2. The highest critical loads of CrN coated DIN 1.2379 steel is obtained among the other steel substrate in terms of normal force (Fn) and these highest load values were obtained in a longer distance except K390 steel.
3. The highest dynamic micro-hardness is obtained from CrN coated DIN 1.2379 steel and then DIN 1.2067, K390, DIN 1.7225 and 1.2842 sequentially.
4. CrN coatings on DIN 1.2379 and DIN 1.7225 steel substrates have significantly better adhesion strength compare to the other type of steel substrates.
5. High carbon content substrate results in high CrN hardness and elastic module.

As a conclusion. adhesion CrN coating on the surface of DIN 1.2379 and DIN 1.7225 steel is better than that of the other steel substrates in terms of mechanical properties.

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