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Doç. Dr. Mustafa Batuhan KURT

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CHAPTER 1

THERMAL MANAGEMENT OF EPOXY-BASED HYBRID COATINGS: MATERIALS, MECHANISMS AND APPLICATIONS

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1. INTRODUCTION

Epoxy resins are among the thermoset polymers widely used in coating and composite applications due to high adhesion, chemical resistance, mechanical stability, and versatility. On the other hand, the inherently low thermal conductivity of neat epoxy remains a significant limitation, particularly involving high heat flux, such as electronic systems, electrical machines, battery systems, and aerospace technologies. The limited ability of epoxy to dissipate accumulated heat can lead to localized temperature increases and adversely affect the durability of coating and substrate materials.

Enhanced thermal conductivity integration fillers into the epoxy matrix has been widely investigated as an effective approach to overcome this limitation. Carbon-based, ceramic, metallic, and systems of hybrid fillers with various morphologies, diameters of particles, and surface characteristics have been employed to enhance the thermal conductivity of epoxy composites. Previous studies have showed that the incorporation of thermally conductive fillers improve heat-transfer capability however, the efficacy of the approach relies on strongly on filler type, concentration, dispersion, morphology, and interfacial interactions within the polymer matrix.

Recent works have demonstrated a distinct shift from single-component filler systems toward multi-component, hybrid, and hierarchical structures. The combined use of fillers with different sizes, geometries, and thermal conductivities has been shown to promote the formation of more continuous and efficient thermal conduction pathways within the epoxy matrix. In particular, the synergistic effects arising from the combination of micro and nanoscale fillers have attracted considerable attention because it can facilitate the development of interconnected thermal networks while reducing the limitations associated with individual filler systems.

In modern engineering applications involving high power density, efficient thermal management is critical for ensuring system reliability, durability, and performance. Accordingly, thermally conductive epoxy-based composites have been increasingly investigated for potential applications in aerospace structures, electrical machinery, and battery systems. In addition to experimental investigations, computational modelling and optimization approaches have been employed to predict thermal performance and identify suitable material compositions and processing parameters. These approaches provide opportunities to reduce experimental effort and establish relationships between filler characteristics, composite structure, and thermal performance.

2. THERMAL CONDUCTION MECHANISM IN EPOXY-BASED SYSTEMS

Epoxy resin was one of the fundamental materials used in the coating industry due to versatile applications and superior performance characteristics. It was regarded as an indispensable component of modern industrial coatings because of its excellent adhesion properties, chemical resistance, and mechanical stability. The intrinsically low thermal conductivity of neat epoxy was considered a significant limitation such as electronic systems, electrical machinery, and thermal management in aerospace systems. Neat epoxy was inherently a thermal insulator. Thermal conductivity of neat epoxy was generally limited due to irregular molecular structure (Yang et al., 2025). This restriction was often associated with the formation of localized hot spots, which could accelerate the degradation of both the coating and the components beneath the coating. Consequently, an industrial need was identified for thermal coatings that could bridge the gap between high protection and high heat dissipation capacity. The primary strategy utilized to improve the thermal conductivity of epoxy was the incorporation of highly thermally conductive fillers into the system. The effectiveness of a filler was determined by its intrinsic thermal conductivity, aspect ratio, dispersion state, and the quality of the interface formed. High filler loadings were often associated with a significant increase in viscosity, deterioration of surface quality, and a reduction in mechanical toughness. Therefore, a complex optimization problem was encountered by materials scientists (Xiao & Du, 2016).

In non-metallic materials such as epoxy, heat transfer was primarily governed by phonons, which were quantized lattice vibrations. In a material with a highly ordered crystalline structure, phonons were able to travel over long distances with limited scattering, and consequently, high thermal conductivity was achieved. In contrast, intense phonon scattering was caused by the amorphous and highly cross-linked structure of epoxy resin at molecular bonds and connection points. When fillers were incorporated into the epoxy matrix, a network was intended to be formed through which phonons could be transported preferentially through the highly thermally conductive filler rather than through the low thermal conductivity polymer. However, two primary sources of phonon scattering were introduced during the process. First, in-filler scattering was observed, in which defects within the filler itself could hinder heat flow. Second, interfacial thermal resistance was generated at the boundary between the filler and the epoxy matrix (Figure 1).

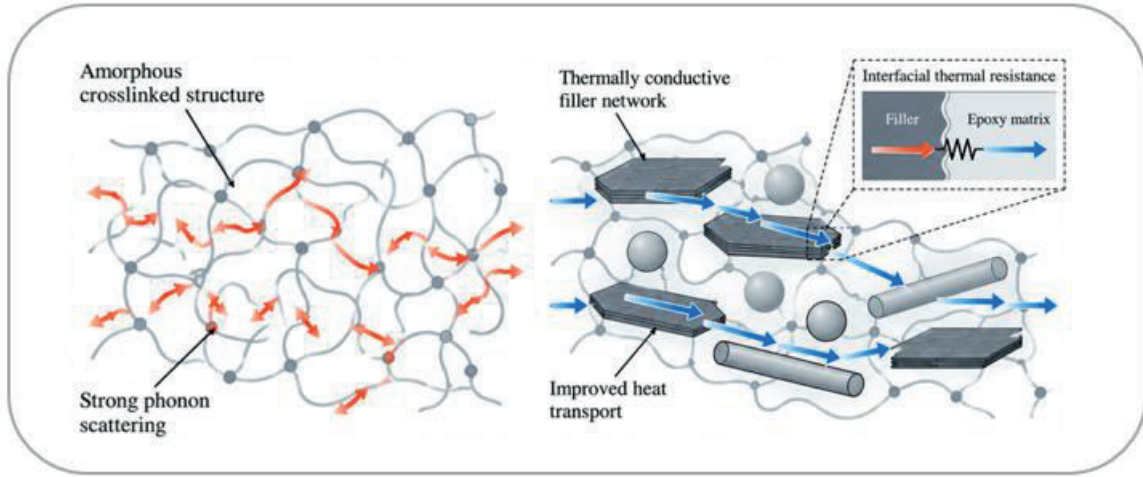


Figure 1. Thermal transport mechanism in epoxy-based systems

A significant difference between the phonon vibrational modes of the organic matrix and the inorganic filler was considered to form a barrier to heat transfer (Ruan et al., 2023). Therefore, optimization of thermal conductivity was considered to require a two-fold approach. Fillers with high intrinsic thermal conductivity and high aspect ratios were selected to reduce the number of interfaces, while the interface was simultaneously engineered to reduce interfacial resistance by improving the compatibility between the phonon vibrational modes (Liang et al., 2012).

3. THERMAL CONDUCTIVE FILLER SYSTEMS IN EPOXY COMPOSITES

Carbon-based fillers were preferred due to high thermal conductivity. The thermal conductivity of individual nanotubes or graphene sheets was reported to exceed 3000 W/m·K. Graphene nanoplatelets and graphene oxide (GO) were among the most widely investigated carbon fillers for epoxy coatings (Lv et al., 2022). Graphene nanoplatelets, which were composed of multiple graphene layers, provided a high surface area and high aspect ratio. This feature facilitated the development of conductive networks at reduced filler concentrations compared with spherical particles (Campo et al., 2020).

Studies showed that the addition of graphene nanoplatelets elevated the thermal conductivity of epoxy (Jarosinski et al., 2017). Higher values were obtained with higher filler contents and optimized dispersion. For example, a thermal conductivity of 1.239 W/(m·K) was attained in graphene/epoxy composites, and an improvement over the neat resin was obtained (Yang et al., 2024). Reduced graphene oxide (rGO) was also found to be effective in increasing thermal conductivity, particularly when it was used together with other insulating particles. The results demonstrated the effect of using two-dimensional carbon fillers (Yung et al., 2022).

In addition, reduced GO/epoxy composites were investigated, and it was shown that the reduction process itself could affect the mechanical and thermal characteristics of final coating (Olowojoba et al., 2016). Graphene oxide (GO) was frequently preferred because it could be easily functionalized. The functional groups on the graphene oxide surface that contain oxygen enabled covalent bonds to be formed with the epoxy matrix, and thus interfacial compatibility and dispersion were significantly improved (Liu et al., 2021). However, defects were also introduced during the oxidation process, which reduced the thermal conductivity of the sheets. Therefore, a balance between dispersion and performance was required. Recent studies have focused on the use of fluorinated graphene nanofillers to improve both thermal conductivity and dielectric properties (Zhang et al., 2023).

Carbon nanotubes provided one-dimensional heat transfer pathways. Multi-walled carbon nanotubes were widely used as a more cost-effective alternative to single-walled nanotubes. It was reported that the thermal conductivity of neat epoxy was doubled by the addition of multi-walled carbon nanotubes. The main advantage of carbon nanotubes was their ability to bridge the gaps between larger filler particles such as graphene or alumina. In this way, a multiscale network was formed, which reduced the overall thermal resistance of the composite (Xu et al., 2026). Functionalization of multi-walled carbon nanotubes was often required to improve their dispersion. However, in applications where electrical insulation was required, the functionalization process had to be carefully carried out to avoid adversely affecting the electrical properties. Carbon fibers were particularly used in thicker coatings or reinforced plastics. Modified pitch-based carbon fibers functionalized through non-covalent bonding with 1-aminopyrene were used to suppress longitudinal thermal conductivity and direct heat flow in the desired direction. Hybridization of carbon fibers with diamond particles or graphene was also considered an effective strategy for forming dense and closely packed networks (Zheng et al., 2025).

Other carbon-related materials were also developed for specific applications. Nanodiamonds provided high thermal conductivity and electrical insulation, and materials could be applied as coatings over other fillers, such as carbon nanotubes, to enhance heat transfer while maintaining dielectric properties (Zhao & Jiang, 2017). MXenes, which were two-dimensional transition metal carbides, exhibited high metallic conductivity and hydrophilic surfaces. Due to unique planar morphology, MXenes were shown to significantly increase the thermal conductivity of epoxy composites even at low contents (Kang et al., 2019). Closely packed two-dimensional diamond particle networks interconnected by short carbon fibers were shown to improve both thermal conductivity and electrical resistivity (Zheng et al., 2025).

In applications where electrical insulation was required, carbon fillers were not considered suitable because of their high electrical conductivity. In such cases, ceramic fillers were mainly preferred. Boron nitride (BN) was often referred to as “white graphene” because of its similar layered structure and high thermal conductivity. Unlike graphene, however, BN was a wide-bandgap electrical insulator and was therefore considered suitable for electrical and electronic coatings (Xiao & Du, 2016). Hexagonal boron nitride (h-BN) was the most used form. The best combination of thermal conductivity and dielectric strength was provided by h-BN (Isarn et al., 2019). In addition to thermal stability, BN was combined with flame-retardant coatings such as poly (phosphazene-co-bisphenol A) to improve thermal conductivity (Qu et al., 2017).

Alumina (Al_2O_3) was one of the main filler materials used in industry because of its low cost and easy availability. However, its effectiveness was strongly dependent on particle size and dispersion. 30 nm Al_2O_3 particles were observed to be much more effective than larger particles (Pan et al., 2021). Alumina was also frequently used in hybrid systems, such as $\text{Al}_2\text{O}_3/\text{BN}$ hybrids, to increase filler packing density (Sanchez et al., 2021). The combination of alumina particles with a three-dimensional alumina foam was also developed as a recent strategy to form a continuous ceramic heat-conduction pathway (Wang et al., 2021).

Zinc oxide (ZnO) was another promising filler, particularly in tetrapod-shaped or nanowire forms. The non-spherical geometries were more favorable for forming conductive pathways through mutual contact at lower filler contents (Guo et al., 2018). ZnO-coated silver nanowires were developed to combine the high thermal conductivity of silver with the insulating properties of ZnO. Thus, a material that was both thermally conductive and electrically resistive was obtained (Zhang et al., 2022). Carbon fiber networks used with ZnO nanowires also showed promising results for highly efficient heat transfer (Lin et al., 2024). Silicon carbide (SiC) provided high hardness and good thermal conductivity. It was generally used together with expanded graphene nanoplatelets to form hybrid networks that improved both thermal diffusivity and impact resistance. In some cases, an improvement in impact resistance was achieved. SiC-SiO₂ core-shell nanowire structures were also shown to increase the thermal conductivity of epoxy composites by forming a strong and well-dispersed thermal network (Shen et al., 2017).

Silicon dioxide (SiO₂) generally exhibited lower thermal conductivity than BN or Al_2O_3 however, it was widely used for surface modification. Nano silica was also used to reinforce epoxy floor coatings, and improvements in both thermophysical and mechanical properties were obtained (Nikje et al., 2012).

Metal fillers such as copper and brass were also used in applications although they were less commonly used due to concerns related to weight and corrosion. The irregular shapes of brass and copper fillers were shown to affect the properties of metal-epoxy composites in rapid molding applications (Hussin et al., 2022). Molybdenum disulfide (MoS_2) microspheres were recently added to epoxy composites and were investigated for their ability to modify thermal conductivity and potentially provide lubricating properties (Liu et al., 2022).

One of the most important trends in recent years was the transition from single-filler systems to hybrid and hierarchical structures. The synergy was attributed to the ability of fillers with different shapes and sizes to fill the gaps within the network in the systems. In this way, the number of contact points was increased and the thickness of the polymer layers between the fillers was reduced. In systems containing 0D and 2D fillers, spherical particles such as Al_2O_3 or SiO_2 were used as spacers between large graphene or BN sheets. In this way, overlapping of the sheets was prevented and a more homogeneous network was formed (Xiang et al., 2024). 1D and 2D fillers, such as carbon nanotubes or carbon fibers, were used to bridge the gaps between graphene nanoplatelets, and a more efficient multiscale network was formed compared with single-filler systems (Wang et al., 2023). The combined use of fillers with different morphologies, such as sheets and tubes, was found to improve both toughness and thermal conductivity in epoxy-based systems. A mixture of graphite and silicon carbide microparticles increased the thermal conductivity of epoxy by forming a dense and multiscale network (Mohanraman et al., 2024). A hybrid filler system containing graphite and graphene nanoplatelets was used to obtain epoxy composites with high thermal conductivity by taking advantage of different aspect ratios (Mahanta et al., 2015). Thermally conductive filler systems for epoxy coatings were shown in Figure 2.

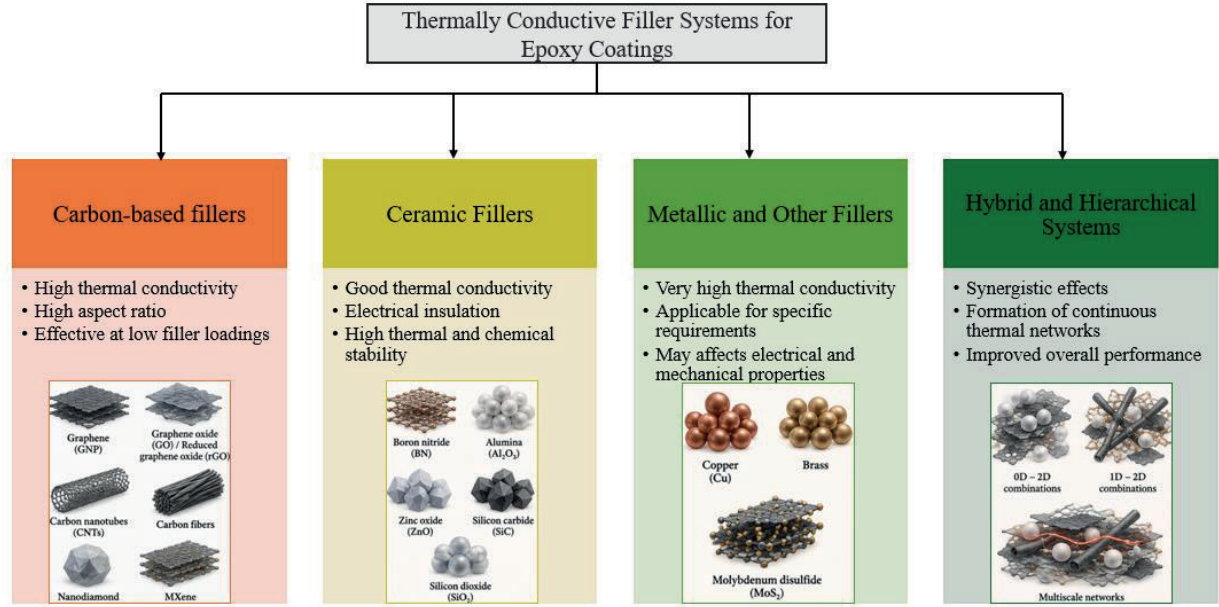


Figure 2. Thermally Conductive Filler Systems for Epoxy Coatings

Hydrophobicity was important for coatings to prevent water-induced degradation and icing in humid environments. In epoxy/graphite oxide coatings, the synergy between hydrophobicity and thermal conductivity was evaluated, and it was shown that a high-water contact angle could be achieved while thermal conductivity was increased (Gholamnejad et al., 2024).

The addition of high amounts of filler often caused coatings to become brittle. Polyacrylic modifiers and flexibilizers were used to maintain impact resistance and fracture toughness (Xu et al., 2019). Three-dimensional networks formed from ceramic or carbon fibers could increase the mechanical strength and hardness of the coating. Studies conducted on BN/epoxy composites focused on achieving high out of plane thermal conductivity together with superior dielectric strength for high voltage applications (Nguyen et al., 2022).

4. APPLICATIONS, MODELLING, AND OPTIMIZATION APPROACHES

Graphene/epoxy coatings with heat dissipation properties were used in the thermal management of spacecraft. Organic/inorganic nanoceramic hybrid resin coatings were applied to motor windings to improve heat dissipation and motor efficiency. Increasing the thermal conductivity of fiber-reinforced polymer composites was considered an important research area for battery enclosures, where fire safety and heat dissipation were of great importance (Gökciler & Green, 2024) (Table 1).

Table 1. Applications of thermally conductive epoxy composites for thermal management

Application area	Thermal management objective	Thermally conductive epoxy system	Function	Reference
Aerospace structural components	Heat dissipation and weight reduction	CNT/epoxy and carbon-based composites	Lightweight structure, high mechanical strength, and enhanced thermal management	Hemath et al. (2020)
High-temperature aerospace components	Thermal protection	Ceramic-filled epoxy composites	Improved resistance to high temperatures and impact while providing thermal management	Hemath et al. (2020)
Electronic packaging	Heat dissipation from electronic components	BN/epoxy and AlN/BN/epoxy composites	Enhanced thermal conductivity combined with electrical insulation	Isarn et al. (2019); Zhang et al. (2019)
Thermal interface materials (TIMs)	Reduction of interfacial thermal resistance	BN/graphite films and	Enhanced through-plane heat transfer and efficient heat spreading	Bashir et al. (2023); Kim et al. (2024)
Electronics	Heat dissipation and thermal spreading	GNP/BNNP hybrid fillers and multiscale filler systems	Formation of efficient and continuous thermal conduction pathways	Wang et al. (2023); Zhang et al. (2019)
Automotive applications	Heat dissipation in powertrain systems	Thermally conductive epoxy composites	Thermal management, lightweight design, and mechanical durability	Zhang et al. (2019); Hemath et al. (2020)
Energy applications	Heat transfer and thermal stability	Thermally conductive polymer composites and boron nitride-based structures	Improved heat transfer and thermal stability in energy-related systems	Samsudin et al. (2022); Tian et al. (2023)

Considering the complexity of hybrid filler systems, computational modelling was considered highly important for predicting performance and optimizing designs. Molecular dynamics simulations were used to investigate the vibrational modes of polymer chains and fillers at the atomic level. The simulations helped to understand the main causes of thermal resistance at the interface and the role of crosslinking density (Liu et al., 2023).

Various predictive models were used to estimate the efficient thermal conductivity of composites, including the Lewis and Nielsen model (Yu et al., 2013), the model suggested by Chu et al. (Galpaya et al., 2012), and random distribution/closest packing models (Yang et al., 2023). Optimization methods such as the Response Surface Methodology and Design of Experiments were used to determine the optimum filler content in terms of both thermal and hydrophobic properties (Nguyen et al., 2022).

5. CONCLUSIONS

The thermal management performance of epoxy-based coatings was extensively investigated owing to the inherently low thermal conductivity of the epoxy matrix. Carbon-based fillers were reported to provide substantial advantages owing to the high intrinsic thermal conductivity and the capacity to form conductive routes even at comparatively low loading levels. For applications in which electrical insulation was required, ceramic fillers such as boron nitride, alumina, zinc oxide, and silicon carbide were considered suitable alternatives.

Since a single-filler system was often unable to simultaneously optimize thermal, mechanical, electrical, and surface properties, increasing attention was given to hybrid and hierarchical structures. The combined use of fillers with different particle sizes and geometries was shown to reduce inter particle voids, increase the number of contact points, and facilitate the formation of more continuous thermal conduction networks. Through this approach, other functional properties, including toughness, dielectric characteristics, and hydrophobicity were also maintained or enhanced.

The development of thermally conductive epoxy systems was considered crucial for a broad variety of engineering uses, including heat dissipation in aerospace vehicles, thermal management of motor windings in electrical machinery, and the control of localized temperature spikes in battery systems. As the structural complexity of hybrid systems was increased, experimental studies were complemented by computational modelling and optimization techniques. Molecular dynamics simulations, theoretical thermal conductivity models, and design of experiments-based optimization methods were employed to investigate filler-matrix interactions and determine optimal compositions.

In conclusion, the primary objective in the design of high-performance thermal epoxy coatings was identified as the establishment of an appropriate balance among thermal, mechanical, electrical, and environmental properties according to application specific requirements. The combined use of well controlled hybrid filler networks, interface engineering, and multiscale modelling approaches was identified as a significant direction for the development of

multifunctional epoxy systems capable of delivering superior thermal performance at lower filler loadings. Future research was expected to focus on optimizing the balance among high thermal performance, mechanical flexibility, and cost-effective manufacturing processes.

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CHAPTER 2

HETEROGENEOUS ELECTRO- FENTON PROCESS: RECENT DEVELOPMENTS AND FUTURE PERSPECTIVES

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INTRODUCTION

The growth of industrial activities has led to the production of wastewater with diverse characteristics. As a result, the presence of organic pollutants that are not easily removed by conventional treatment methods has become a major environmental challenge. In particular, the potential risks posed to aquatic environments even at low concentrations by poorly biodegradable pollutants, such as pharmaceutical compounds, antibiotics, phenolic compounds, pesticides, and dyes, have increased interest in advanced oxidation processes (AOPs). Among these processes, Electro-Fenton (EF) is an effective electrochemical advanced oxidation process that produces hydrogen peroxide (H_2O_2) using electricity. The generated H_2O_2 then reacts with iron species to produce hydroxyl radicals ($\bullet\text{OH}$), which are responsible for the oxidation of organic pollutants. Depending on the cathode and catalyst employed, EF systems can be classified as homogeneous and heterogeneous EF processes. In recent years, increasing research attention has been directed toward heterogeneous EF as a means of overcoming some of the limitations associated with homogeneous EF. In conventional homogeneous EF systems, dissolved $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions participate in the catalytic conversion, while H_2O_2 is generated at the cathode through the two-electron reduction of oxygen. However, using dissolved iron ions can limit the operating conditions of the EF process and cause some practical problems. These include the need to maintain an acidic pH, neutralization after treatment, and the formation of sludge due to iron hydroxide precipitation (Poza-Nogueiras et al., 2018; Sharma and Feng, 2019). The literature reports that the effective operating range for homogeneous EF is generally around pH 2.8–3.5 (Nair et al., 2021).

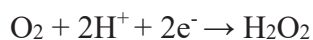
Heterogeneous EF processes have been developed to overcome some of the associated with homogeneous EF by immobilizing iron species on a solid catalyst or a functionalized cathode rather than allowing them to remain freely dissolved in the solution. This approach enables the formation of active sites on the solid phase, where the Fenton reaction can proceed catalytically, while also allowing the catalyst to be separated from the reaction medium and reused after treatment. One of the major advantages of heterogeneous EF is its potential to operate over a broader pH range than homogeneous systems. This feature can reduce the need for post-treatment neutralization and, consequently, help limit the formation of iron hydroxide sludge and the associated treatment problems (Poza-Nogueiras et al., 2018; Nidheesh et al., 2014). In addition, the easy separation and recovery of solid catalysts, their ability to maintain catalytic activity over repeated cycles, and their good storage stability can improve the practical use of heterogeneous EF systems. For this reason, a wide range of iron-based materials have

been studied as heterogeneous EF catalysts, including natural iron minerals such as pyrite, goethite, and magnetite, as well as iron species used alone or supported on porous materials (Babuponnusami and Muthukumar, 2014).

THE BASIC PRINCIPLE AND MECHANISM OF THE HETEROGENEOUS ELECTRO-FENTON PROCESS

The fundamental principle of the heterogeneous EF process involves the electrochemical generation of H_2O_2 through the reduction of oxygen at the cathode, which is continuously supplied with air or oxygen, followed by the activation of H_2O_2 by active metal sites on the solid catalyst to generate highly reactive oxidizing species. The key distinction from homogeneous systems is that the Fenton catalytic conversion is intended to occur predominantly on the solid phase. In this approach, the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox couple is maintained on the solid catalyst, while H_2O_2 is activated at the catalyst surface.

The fundamental reaction taking place at the cathode under a continuous supply of air or oxygen can be expressed as follows:



The H_2O_2 generated at the cathode can subsequently interact with the $\equiv\text{Fe}^{2+}$ sites on the catalyst surface, leading to the formation of $\bullet\text{OH}$ radicals. The reduction of surface $\equiv\text{Fe}^{3+}$ species to $\equiv\text{Fe}^{2+}$ through electron transfer, followed by the activation of H_2O_2 by the $\equiv\text{Fe}^{2+}$ sites, constitutes the basis of the heterogeneous catalytic conversion. This process is generally described in terms of surface-mediated $\text{Fe}^{3+}/\text{Fe}^{2+}$ redox conversion and H_2O_2 activation. In addition, Fe^{2+} leaching may occur under acidic operating conditions. Consequently, a portion of the process may proceed through a homogeneous EF pathway (Figure 1).

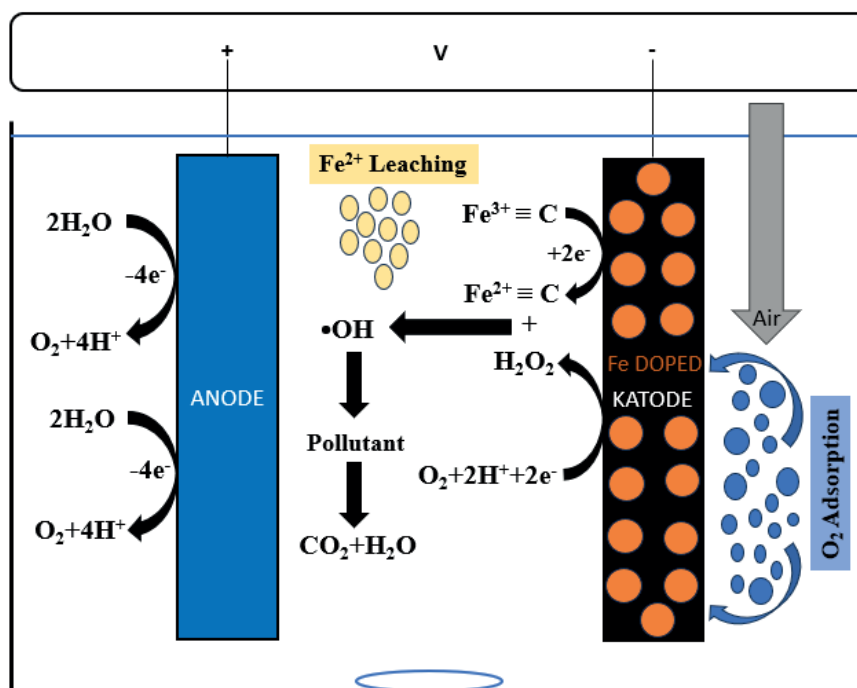


Figure 1. Schematic representation of electrochemical H_2O_2 production and the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox conversion in a heterogeneous EF process.

The mechanism of heterogeneous EF is too complex to be described by a single reaction pathway. In particular, the catalyst structure, surface properties, coordination state of iron, and operating pH can strongly influence the reaction pathway. The generally accepted mechanism of heterogeneous EF is considered to be analogous to that of the conventional homogeneous Fenton reaction. Under acidic conditions, partial dissolution of the catalyst may release Fe^{2+} and Fe^{3+} into the solution, allowing these species to contribute to the homogeneous Fenton reaction. Therefore, it may not be appropriate to attribute the high removal efficiencies observed in some heterogeneous systems entirely to surface catalysis. In particular, iron leaching may increase at pH values below 3, causing the process to acquire a partially homogeneous character through the contribution of dissolved iron species (Hu et al., 2011; Wang et al., 2013). In contrast, under neutral and alkaline conditions, the role of surface catalysis may become more pronounced due to the reduced solubility of dissolved Fe^{3+} species. Therefore, when evaluating heterogeneous EF systems, it is important to consider not only pollutant removal efficiency but also the dissolved iron concentration, catalyst stability, and extent of Fe leaching.

Since a substantial proportion of catalytic reactions in heterogeneous systems takes place at the catalyst surface, the surface area, pore structure, distribution of active sites, and electrical properties of the catalyst can directly influence process performance. Therefore,

developing a material with a high iron content alone is not sufficient; the catalyst should also possess a structure that enables efficient H_2O_2 activation, facilitates electron transfer, and ensures the accessibility of active sites to the reaction medium (He et al., 2016; Zhu et al., 2019).

MATERIALS FOR CATALYSIS AND H_2O_2 GENERATION IN HETEROGENEOUS ELECTRO-FENTON SYSTEMS

One of the key factors determining the performance of heterogeneous EF processes is the chemical and physical properties of the catalyst. In catalyst selection, not only high catalytic activity but also chemical stability, low metal leaching, reusability, an appropriate surface area, good electron conductivity, and cost-effective synthesis should be considered together. In addition, long-term stability of the catalyst under reaction conditions and its easy separation after use are important for the practical applicability of the process (Ganiyu et al., 2018; Nidheesh, 2015; Guo et al., 2023).

Iron oxide and natural iron minerals

One of the most widely investigated catalyst groups in heterogeneous EF processes comprises iron oxides and naturally occurring iron-bearing minerals. Materials such as magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite, and pyrite can be used to activate H_2O_2 . A major advantage of these materials is that they contain iron active centers within their solid structure, enabling Fenton reactions to occur. In one study, FeS_2 was reported to generate superoxide anions on its surface, which accelerated the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox conversion and promoted H_2O_2 decomposition, thereby enhancing hydroxyl radical generation for diclofenac degradation through a heterogeneous EF-like process. In addition, FeS_2 was found to be highly effective in catalyzing H_2O_2 decomposition for diclofenac degradation over a relatively broad pH range (3–9) (Yu et al., 2020). In another study using natural pyrite as the catalyst, an azo dye with a concentration of 175 mgL^{-1} (initial TOC: 100 mgL^{-1}) was almost completely mineralized within 8 h using a BDD anode and carbon felt cathode. The process was shown to be more effective than the conventional EF process under the same operating conditions (Labiadh et al., 2015).

Fe_3O_4 is one of the most widely investigated iron-based catalytic materials in heterogeneous Fenton processes because its crystal structure contains both Fe^{2+} and Fe^{3+} ions. This structural feature provides an important advantage by facilitating the availability of the iron species required for the Fenton reaction and sustaining the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox conversion. In addition, the magnetic properties of Fe_3O_4 can enable the catalyst to be easily separated from the reaction medium after treatment using an externally applied magnetic field, allowing its

subsequent reuse. However, particularly in nanoscale Fe_3O_4 particles, interparticle agglomeration may occur due to magnetic dipole–dipole interactions and van der Waals forces, which can reduce the effective surface area of the catalyst. In one study, an Fe_3O_4 @carbon composite was used as a cathode together with a carbon aerogel in an EF process. Approximately 95% of dimethyl phthalate was removed within 120 min, with 65% mineralization achieved (Wang et al., 2016). In another study, the degradation efficiency of methylene blue using an Fe_3O_4 -coated stainless steel mesh cathode under neutral conditions was reported to be higher than that obtained under acidic conditions when only a stainless steel mesh was used as the cathode in the EF system (Choe et al., 2021). In a more recent study, Fe_3O_4 synthesized by a controlled coprecipitation method was used as a recyclable solid iron source. After 90 min, magnetic-field assistance increased methylene blue color removal at pH 3 from 14.2% to 46.0% (Hernández-Rodríguez et al., 2026).

Iron oxide-based nanomaterials have attracted considerable attention in a wide range of applications, including environmental engineering and energy technologies, owing to their distinctive magnetic, surface, and structural properties. In particular, Fe_3O_4 offers important advantages due to its superparamagnetic behavior and high magnetic susceptibility, which can facilitate its magnetic recovery from the treatment medium after application. Moreover, the Fe_3O_4 surface can be modified with different functional groups or components, allowing its surface characteristics and interactions with pollutants to be tailored for specific applications. Such surface modifications can also contribute to enhancing the catalytic performance of Fe_3O_4 . The combination of magnetic recoverability, surface functionalization, and tunable catalytic properties makes Fe_3O_4 and Fe_3O_4 -based nanomaterials promising candidates for environmental applications, particularly in water and wastewater treatment (Shah et al., 2025).

Supported and composite iron-based catalysts

To overcome some of the limitations associated with bare metal oxides, immobilizing iron species on various support materials, such as carbonaceous materials, clay minerals, and chitosan, has become a widely adopted strategy. These supports serve functions beyond simply providing a matrix for iron species. They can help suppress particle agglomeration, increase the accessible active surface area, facilitate electron transfer, and improve the mechanical stability of the catalyst.

Clay minerals have been used as supports for iron oxide immobilization because of their low cost and inherent adsorption capacity. For example, Fe₂O₃-modified kaolin has been investigated as an alternative heterogeneous catalyst for the removal of enoxacin. Carbon-based supports are also particularly attractive for heterogeneous EF applications because of their high surface area, electrical conductivity, and chemical stability.

Carbon aerogel, carbon felt, graphite felt, activated carbon, and graphene can be employed both as cathode materials that promote H₂O₂ generation and as support materials for the immobilization of iron species. In particular, the dispersion of iron species within carbon-based structures can help limit nanoparticle agglomeration by reducing direct contact between individual nanoparticles. An iron–carbon composite was applied as a heterogeneous electro-Fenton catalyst for treating dyeing wastewater, with Reactive Red X-3B selected as the model pollutant. Among the catalysts examined, the iron–carbon material exhibited the highest removal performance, reaching $99.63 \pm 1\%$ for color and $79.86 \pm 2\%$ for COD. Its catalytic stability was also assessed through repeated EF cycles. After five consecutive conversions, the composite maintained its catalytic performance without any appreciable loss in activity, indicating good reusability (Yao et al., 2021).

In another study, Fe- and N-doped biochar was employed as a cathodic catalyst in an EF system. The material was reported to promote the two-electron reduction of oxygen, thereby facilitating the in situ generation of H₂O₂, followed by its activation and the formation of •OH and •O₂[−] species. The catalyst also exhibited good reusability while maintaining its catalytic activity over successive conversions (Li et al., 2021). An undivided heterogeneous EF reactor incorporating a Ti mesh cathode and a boron-doped diamond (BDD) anode was employed, with Fe₃O₄–chitosan serving as the heterogeneous catalyst. The system enabled in situ H₂O₂ production at the cathode while facilitating the Fe³⁺/Fe²⁺ redox conversion. Consequently, the generated Fe²⁺ reacted with H₂O₂ to produce hydroxyl radicals (•OH), which effectively contributed to phenol degradation, achieving a removal efficiency of 93%. (Pujol et al., 2020). In one study, approximately 94.83% removal of Acid Blue 25 was achieved within 90 min, while COD and TOC removal reached 74.19% and 61.53%, respectively. The catalyst maintained its activity after five reuse cycles and showed low iron leaching. The heterogeneous EF process also achieved about 16% higher removal than the homogeneous EF process (TaheriAshtiani and Ayati, 2022). In another study, an Fe-bentonite catalyst was used for the removal of malachite green and methylene blue. Under optimum conditions, removal efficiencies of 98.06% and 91.14% were obtained, respectively, with iron leaching below 0.1

ppm. The heterogeneous EF process showed better performance than EF alone and bentonite adsorption (Thwaini et al., 2026).

Fe-based nanomaterials have attracted considerable attention in water treatment owing to their ability to remove contaminants through a combination of magnetic separation, adsorption, and catalytic processes. Fe_3O_4 nanoparticles and Fe_3O_4 -based nanocomposites can be synthesized using several methods, such as co-precipitation, green synthesis, hydrothermal treatment, sol–gel synthesis, and electrochemical techniques (Shah et al., 2025). Co-precipitation is particularly common due to its straightforward procedure, low cost, good synthesis efficiency, and suitability for controlling the size and morphology of the resulting particles (Liu et al., 2020; Patil et al., 2021; Zaqa et al., 2024). The nature of the base used during synthesis can also influence the morphology and structural characteristics of the resulting particles (BaAbbad et al., 2022).

Green synthesis has emerged as an environmentally benign alternative, particularly through the use of biological resources such as plant extracts as reducing and stabilizing agents (Ramesh et al., 2018; Yusefi et al., 2021). Hydrothermal and solvothermal routes, on the other hand, are advantageous for obtaining well-defined crystalline structures while offering high purity and good reproducibility under relatively moderate temperature conditions (Xie et al., 2009; Acharyya et al., 2014; Reith et al., 2018). The sol–gel approach is also of interest because it enables the production of materials with high purity and good chemical homogeneity (Castro et al., 2002; Asgari et al., 2019). During green synthesis, the characteristics of the resulting nanoparticles are strongly influenced by synthesis conditions, particularly the pH, amount of plant extract, and solvent composition, which can alter their physicochemical and magnetic properties. Beyond their individual application, Fe_3O_4 -based nanoparticles can be incorporated into hybrid treatment systems, including photocatalytic processes, membrane filtration, and coagulation, to improve overall treatment performance. However, despite their promising laboratory-scale results, further pilot-scale investigations are required to assess their long-term stability, operational performance, and practical feasibility before implementation in full-scale water treatment applications (Shah et al., 2025).

THE ADVANTAGES AND CURRENT LIMITATIONS OF THE HETEROGENEOUS ELECTRO-FENTON PROCESS

Catalyst Recovery

One of the major advantages of heterogeneous EF systems is the ability to retain iron species on a solid catalyst, allowing the catalyst to be readily separated from the reaction medium and potentially reused under appropriate operating conditions. This feature can minimize the formation of iron-containing sludge associated with dissolved iron ions and simplify the separation and post-treatment steps following the EF process. Consequently, catalyst recovery and reuse can contribute to improving the operational sustainability and overall practicality of heterogeneous EF systems (Poza-Nogueiras et al., 2018; Ganiyu et al., 2018).

Operation over a Wider pH Range

Compared with conventional homogeneous Fenton systems, certain heterogeneous EF processes can maintain catalytic activity over a relatively broader pH range. This operational flexibility may reduce the need for extensive pH adjustment before or after treatment, thereby lowering the consumption of additional chemicals. As a result, the ability to operate under less restrictive pH conditions can contribute to reducing operating costs and improving the practical feasibility of heterogeneous EF systems (Poza-Nogueiras et al., 2018; Ganiyu et al., 2018).

Magnetic Catalysts and Immobilization

Magnetic materials, particularly Fe_3O_4 -based catalysts, offer the advantage of being readily separated from the treated solution by means of an external magnetic field, thereby improving catalyst recovery and potential reusability. Another approach involves anchoring iron-containing catalytic species directly onto the cathode, which minimizes catalyst loss from the reaction system and allows the catalytic material to remain available for repeated electro-Fenton cycles. Nevertheless, the potential detachment of the catalyst from the electrode surface and the associated loss of catalytic activity should be considered, particularly during prolonged operation (Ganiyu et al., 2018; Muzenda and Arotiba, 2022).

Iron Leaching

Iron leaching, which refers to the release of iron species from the solid catalyst into the aqueous phase, represents one of the main challenges associated with heterogeneous EF

processes. The extent of iron dissolution may increase under acidic operating conditions, potentially compromising catalyst stability and promoting the occurrence of homogeneous Fenton reactions in the solution. In addition, the release of iron into the treated effluent may result in residual iron concentrations, which can create an additional requirement for downstream treatment or separation (Ganiyu et al., 2018; Muzenda et al., 2023).

Catalyst Agglomeration

The agglomeration of nanoscale Fe_3O_4 and Fe_2O_3 particles can reduce the available active surface area and consequently impair catalytic performance. Immobilizing the catalyst onto suitable supports, such as carbon materials, biochar, clay, or electrode surfaces, can help mitigate particle aggregation. However, if the interaction between the catalyst and the supporting material is insufficiently strong, detachment of the catalyst may occur, resulting in catalyst loss and a decline in process performance (Ganiyu et al., 2018; Muzenda and Arotiba, 2022).

Cost and Scale-Up

The preparation of nanocatalysts and functionalized electrodes used in heterogeneous Electro-Fenton processes can increase overall costs because of the complexity of the synthesis and fabrication procedures. In addition to material preparation, factors such as electricity consumption, electrode replacement, catalyst regeneration, and waste management contribute to the overall operating cost of the process. Therefore, achieving high pollutant removal efficiencies at the laboratory scale does not necessarily guarantee economic feasibility at larger scales. Comprehensive technical and economic assessments are consequently required to determine whether the performance achieved under laboratory conditions can be effectively translated to pilot- and industrial-scale applications (Ganiyu et al., 2018; Shokri et al., 2023).

FUTURE STUDIES AND CONCLUSIONS

The heterogeneous EF process offers important advantages over conventional homogeneous EF systems, particularly in terms of catalyst recovery, reduced iron sludge formation, and the possibility of operating over a broader pH range. However, further improvements are needed to achieve high catalytic activity together with low iron leaching, long-term stability, reusability, and low-cost material synthesis. In particular, the development of efficient and economically feasible cathode materials, improved H_2O_2 generation and

utilization, and a better understanding of the reaction mechanisms at higher pH values remain important research priorities.

Future studies should also focus on the performance of heterogeneous EF catalysts in real wastewater, long-term operation, and continuous-flow reactor systems. Catalyst synthesis, electrode configuration, and reactor design should be optimized together to improve process efficiency while reducing energy and operating costs. In addition, techno-economic evaluation and scale-up studies are required to determine the practical feasibility of heterogeneous EF systems.

Overall, the future development of heterogeneous EF should consider not only pollutant removal efficiency but also catalyst cost, stability, Fe leaching, energy consumption, reusability, and long-term operational performance. Addressing these factors simultaneously will be essential for advancing heterogeneous EF toward a sustainable, economically viable, and practically applicable wastewater treatment technology.

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CHAPTER 3

ROLE OF ZINC BORATE AND TALC IN TAILORING THE PROPERTIES OF AUTOMOTIVE POLYMERS

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1. INTRODUCTION

There were many commercially used flame retardants in literature. Some of those were compounds consisting of elements such as boron, phosphorus, antimony, chlorine, bromine [1]. The use of antimony trioxide and antimony trioxide-halogen mixtures among these compounds was prohibited due to the formation of toxic fumes during combustion [2]. In studies, various flame retardants and minerals were added to different polymer materials and the properties of polymers such as combustion and physical strength were investigated. The findings demonstrated that zinc borate into the polymer matrix significantly enhanced the thermal resistance of polyvinyl alcohol, indicating its effective role as a stabilizing additive [3].

In another study, the flame-retardant performance of various zinc borate compounds was systematically examined in polyurethane foam systems. The results highlighted the effectiveness of different zinc borate formulations in enhancing the fire resistance of polyurethane foam, demonstrating their potential as efficient flame-retardant additives. The results highlighted the effectiveness of different zinc borate formulations in enhancing the fire resistance of polyurethane foam, demonstrating their potential as efficient flame-retardant additives. After the analysis, it was observed that the maximum decomposition temperature of the polyurethane foam increased [4]. By different researchers, zinc borate was synthesized and added to PET together with other flame-retardant materials, and its combustion properties and impact resistance were investigated. In results, it was seen that PET containing zinc borate had higher flame and impact resistance than pure PET [5].

In a different study, zinc borate and colemanite added polypropylene composites were produced and characterized. Antioxidant was used in the study to prevent oxidation. The rheological properties of the produced composites were determined. LOI analysis was performed on composites with impact, tensile and three-point bending tests [6]. Several studies have documented the preparation of zinc borate modified with surfactants and evaluated its impact on the physicochemical properties of polyethylene terephthalate (PET). Surface-treated zinc borates were utilized in conjunction with boron phosphate as flame-retardant agents in PET formulations. The fire-retardant effectiveness of the developed composites was quantified through limiting oxygen index (LOI) measurements, which confirmed the beneficial contribution of these additives to fire resistance [7].

Several reports in the literature have also addressed the oxidative stability and flame-retardant behaviour of polyurethane–zinc borate composite systems. It has been demonstrated that zinc borate substantially enhances the oxidation resistance of the pristine polymer, as initially indicated by oxygen induction time (OIT) analyses and subsequently corroborated by long-term weathering chamber experiments. Comparative evaluations revealed that zinc borate–filled polyurethane exhibited markedly improved performance relative to unfilled polyurethane, evidenced by prolonged oxygen induction times and superior durability under environmental exposure conditions [8].

Thermogravimetric analysis results indicated that the presence of both fillers significantly modified the thermal degradation behaviour of the PVC matrix. Cone calorimetry measurements further showed that wood flour contributed positively to the combustion behaviour of PVC composites, although its effect on reducing smoke generation was relatively limited [9]. In a related study, thermogravimetric data revealed that zinc borate modified the degradation mechanisms and improved the high-temperature thermal stability of these composite systems [10].

In a separate research approach, polyurethane/zinc borate nanocomposites were fabricated and comprehensively characterized. Within this framework, zinc borate particles were surface functionalized using poly(propylene glycol) phosphate ester nanoparticles in combination with oleic acid. The addition of a small quantity of oleic acid was found to markedly enhance the dispersion of zinc borate nanoparticles throughout the polyurethane matrix. In addition, both oleic acid and surface-modified zinc borate exerted a significant positive influence on the thermal stability of polyurethane [11].

Moreover, nano-scale zinc borate was synthesized and incorporated into polyvinyl chloride composites to assess its influence on material performance. The findings indicated that the inclusion of nano zinc borate did not result in any notable deterioration of the mechanical properties of PVC-based composites [12]. Likewise, zinc borate–containing polyurethane films were produced and systematically characterized in various studies, which demonstrated that the presence of zinc borate did not adversely affect the mechanical performance of the polyurethane films, further highlighting its potential as an effective functional additive [13].

Some researchers studied the flame retardant and smoke inhibiting effect of zinc borate. The best results were obtained by adding 10% by weight of zinc borate, 40% by weight of aluminium trihydrate and 8% by weight of trichloro propyl phosphate [14]. In the literature, zinc borate was synthesized and characterized in different studies and the flame-reducing effect of polystyrene was investigated. The compounds exhibited good flammability behaviour in relation to the LOI and became suitable for green flame retardants [15].

Different researchers investigated the rheological properties of zinc borate doped polypropylene. In the study, polypropylene granules were produced by adding zinc borate at different rates to powdered polypropylene. As a result of the study, it was observed that the viscosity value increased as the zinc borate additive ratio increased, but the viscosity decreased as the temperature and pressure increased. In addition, it was found that the shear rate decreased with increasing additive ratio, but the shear rate increased with increasing temperature and pressure [16]. Other researchers experimentally examined the influence of zinc borate on the combustion behaviour of polymeric composite materials. In their study, the flammability characteristics of the composites were evaluated by applying a zinc borate-based surface coating layer with varying thicknesses. The experimental analyses indicated that the most effective fire-resistant performance was achieved when the zinc borate protective layer constituted approximately 30% of the coating thickness and when the exposure interval was relatively long, leading to improved flame-retardant behaviour of the composite materials [17]. Optimization methods were ways to minimize the number of experiments by using orthogonal arrays while examining the effects of variables on the process. It contributed to the improvement of product quality while defining the appropriate control parameters to obtain the most appropriate results [18, 19].

Taguchi, one of the optimization methods, was frequently used in many engineering applications to reach the best experimental design and to optimize the parameters well [20]. Taguchi method, which aimed to reduce the variance on the output, simplified the classical experimental design by using orthogonal arrays. Taguchi's use of signal and noise ratios for variance reduction was a first in experimental design [21]. The effects of the selected parameters were minimized. Orthogonal arrays were used while these parameters are blocked [22]. Tolerance design consists of studies to be carried out if the expected target could not be reached because of parameter design. By using the values reached, the losses caused by the deviation of the product from the target value were found. A wide tolerance range was generally

not preferred [23-25]. Also, there were various studies on boron types and composite materials used in automotive industry [26-28].

In this study, unlike other studies in the literature, flame retardancy and durability in plastics, which were important for industry, were studied. Zinc borate and talc were added to the plastic materials used in the automotive industry, and the Taguchi optimization method was used to increase efficiency and define the optimum parameters. To evaluate the structural, thermal, and mechanical characteristics of the composites, various analyses and tests were applied.

2. EXPERIMENTAL

2.1. Materials

Polystyrene (PS) and polyvinyl chloride (PVC) were provided from Sigma-Aldrich as analytical reagent. Zinc Borate and dichloromethane were taken from Melos A.Ş. and Merck from Turkey, respectively. Talc was used as powder with high purity.

2.2. Method

First, polymers were dissolved in dichloromethane and made ready to mix with other materials. Zinc borate and talc were added at certain ratios between 5-20% (w/w) over 10 g of polymer. The mixture was stirred in a magnetic stirrer at temperatures determined between 50-80°C and for a certain period between 5-20 minutes, and the resulting mixture was kept in an oven to remove the solvent. As a result of the process, zinc borate polymer composites were obtained (Figure 1). Each of these processes was repeated for polyvinyl chloride and polystyrene, according to the Taguchi optimization method, as 4 level 4 parameters, 16 experiments for a total of 32 experiments.

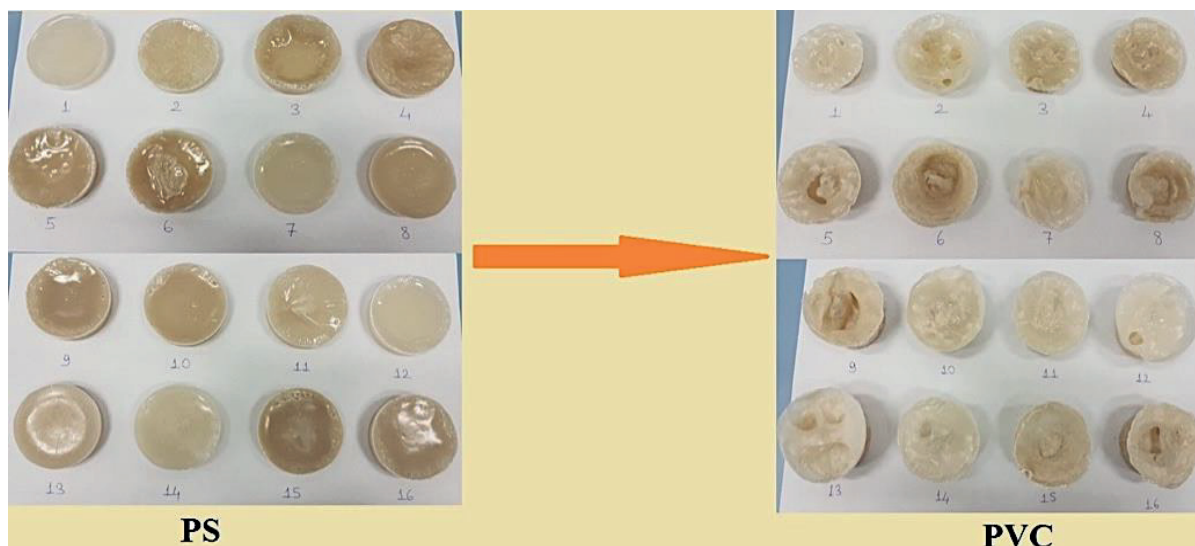


Figure 1. Taguchi Experimental Sets for PS and PVC

With the Perkin Elmer branded Spectrum 100 model FT-IR device, which could analyse the functional groups and bonds in the structure of solid or liquid substances, the raw materials used, and the structures of the products obtained were determined. Thermogravimetric analysis involved the measurement of changes with controlled heating conditions. It was a method in which the weight of the sample was continuously determined by heating the sample, usually at a linear rate. The Oxygen Induction Test was the analysis in which the decay time of the material was determined by giving oxygen to the material under high temperature. SII Nanotechnology branded SII6000 Exstar TG/DTA 6300 model device, which could be used to evaluate mass loss depending on temperature, mass loss depending on time, energy transitions depending on temperature and time, reaction rate and degradation tests, water and ash content, decomposition, oxidation, and heat resistance.

Decomposition temperatures of the raw materials, along with the degradation behaviour of the resulting products, were assessed through the analysis. Analysis provided to determine the morphology of the products obtained in the Zeiss brand EVO-LS 10 model device, which could be used in areas such as determining the composition of minerals, detecting the defects of materials, determining the surface properties, particle size and morphology of the materials.

Tensile and compression devices measured the strength of the material by applying axial force at a controlled speed. In the tensile test, the specimen was pulled axially and, in the test, the elastic limit of the specimen, the maximum tensile stress, yield point, elongation and cross-

sectional contraction were measured. The difference of the compression test was that the direction of the applied axial force was in the opposite direction of the tensile force. Mohr&Federhaff Ag branded test device was tested to examine the material strength.

3. RESULTS AND DISCUSSION

3.1. Taguchi method results

The fundamental principle, which was employed as an optimization approach, is to minimize the number of required experiments using orthogonal arrays while systematically evaluating the effects of process variables. Taguchi method's use of signal (S) and noise (N) ratios for variance reduction was an important point in the experimental design. While evaluating the S/N ratios, the most suitable one was determined from among the small value good, nominal value good and large value good options.

In this study, Experiments were structured according to a Taguchi L16 orthogonal array, with four parameters considered at four separate levels. Optimization parameter levels (settings) of the experiments and Signal/Noise (S/N) values of PS and PVC were given in Figure 2 and Table 1 [29].

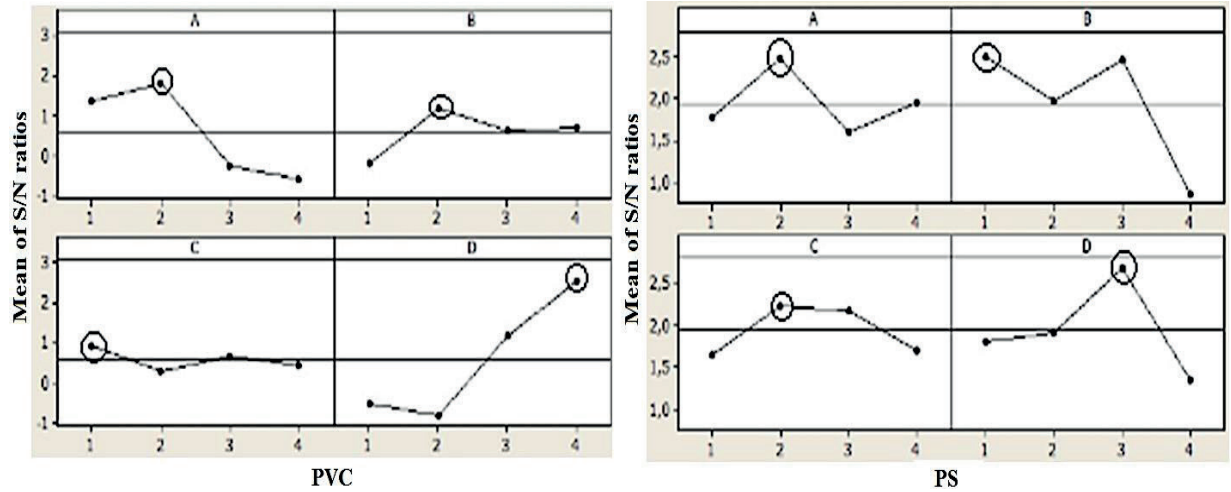


Figure 2. Graphs for S/N values for parameters

Table 1. Experimental parameters (variables) with defined levels

Experimental variables	Setting 1	Setting 2	Setting 3	Setting 4
Zinc Borate (%)	5	10	15	20
Temperature (°C)	50	60	70	80
Stirring Time (min)	5	10	15	20
Talc (%)	5	10	15	20

Based on the impact strength results calculated according to the measurements given by the compression tensile device, according to the principle of 'The biggest is the best'; It was seen that experiment number 6, in which the second level for the zinc borate additive ratio, the second for the temperature, the first for the mixing time and the fourth level for the talc was used, gave the optimum result for the composite material prepared with polyvinyl chloride.

3.2. Thermal analysis results

When the TG analysis result for PS was examined in detail, a mass loss of 4.8% is observed between 100°C and 200°C, and a secondary mass loss of 0.6% between 200°C and 350°C. Finally, a mass loss of 73.5% was observed between 350°C and 550°C. There were three different endothermic peaks in the DTA curve. In general, when the two curves were compared, the total mass loss was 98.9% in pure PS, while the total mass loss in the prepared composite is 78.9%. That is, the degradation of the material was reduced by the added additives. In addition, it was seen that the decomposition takes place at higher temperatures in the prepared composite compared to pure PS.

In the TG curve obtained for PVC, mass loss was observed in 4 steps. These were 4.8% between 110-180°C, 5.6% between 180-220°C, 32.6% between 220-375°C and 13% between 375-580°C. In the DTA curve, on the other hand, an endothermic 4 peak was observed. When the two curves were compared, it was determined that while the mass loss was 93.1% in pure PVC, it was 56.5% in composite material. It was concluded that the degradation of PVC decreased with the added additives.

For the OIT analysis of the prepared PS composite, the composite material was heated up to 193°C by giving nitrogen to the environment. After 193.3°C, oxygen was given to the environment. The material did not burn up to 204.6°C for 44.27 minutes. It has been observed

that the prepared composite material burns later in an oxygenated environment compared to pure PS, that is, it is more durable. For the OIT analysis for PVC, the material was heated up to 193°C in a nitrogen environment. From 193.2°C, oxygen gas was given to the environment. PVC did not burn for 32.9 minutes. It started to burn from 204.4°C. The material was heated up to 190.8°C in a nitrogen environment. From 190.8°C, oxygen gas was given to the environment. PVC composite did not burn for 1.8 minutes. It started to burn from 198.8°C (Figure 3) [29].

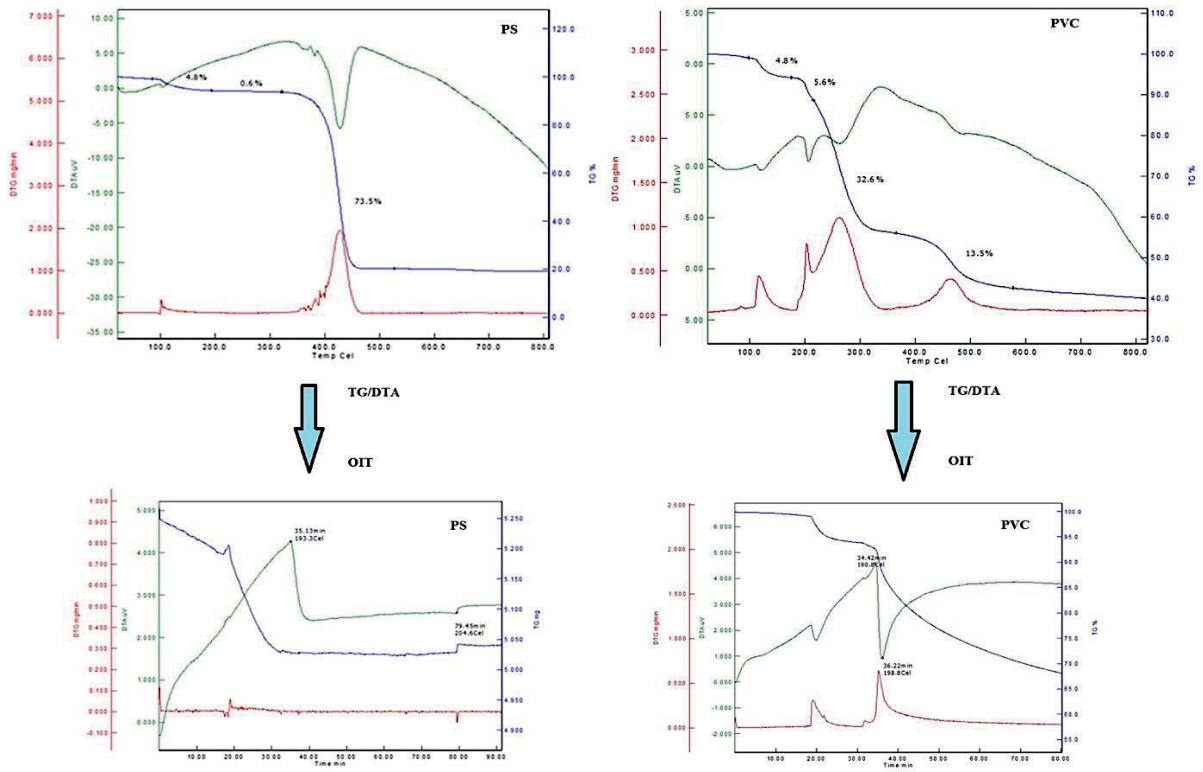


Figure 3. TG/DTA and OIT analysis results for PS and PVC

3.3. Physical analysis results

Viscosity measurements were made three times for the optimum two sets determined by the Taguchi method from the prepared PS and PVC composites, and the averages were calculated. In experiments, a total of 32 of polymer composites, 16 of PVC and 16 of PS, were obtained. The obtained polymers were analysed in the Compression-Tension Tester and the compressive strength values were calculated, comparatively (Table 2) (Table 3) [29].

Table 2. Compressive strength values of prepared PS and PVC composites

No	PVC (Newton)	Diameter (m)	Compressive strength (N/mm ²)	PS (Newton)	Diameter (m)	Compressive strength (N/mm ²)
1	1000	0,03616	0,881	1800	0,05156	1,235
2	1130	0,03763	1,063	1930	0,05053	1,352
3	1190	0,03695	1,282	1990	0,05045	1,570
4	1255	0,03645	1,566	2055	0,05065	0,861
5	1200	0,03808	1,254	2000	0,05065	1,397
6	1265	0,03893	1,725	2065	0,05071	1,297
7	1030	0,03703	1,107	1830	0,05062	1,439
8	1150	0,03802	0,963	1950	0,05190	1,197
9	1250	0,04313	1,154	2080	0,05057	1,310
10	1220	0,03958	0,982	2020	0,05062	1,271
11	1160	0,03998	0,924	1960	0,05272	1,076
12	1050	0,03970	0,842	1850	0,05072	1,162
13	1175	0,04301	0,725	1975	0,05023	1,391
14	1090	0,04029	0,957	1890	0,05424	1,110
15	1300	0,04052	1,022	2100	0,05237	1,277
16	1240	0,04046	1,084	2040	0,05240	1,240

Table 3. Viscosity values of prepared composite materials

	Plastics viscosity containing PS (cP)	Plastics viscosity containing PVC (cP)
Measurement-1	184	598
Measurement-2	185	600
Measurement-3	186	601
Average	185	600

3.4. SEM analysis results

When the morphological structure for PS was examined, it was seen that zinc borate was distributed in masses in the structure. For PVC, it was observed that zinc borate agglomerated in the structure (Figure 4) [29].

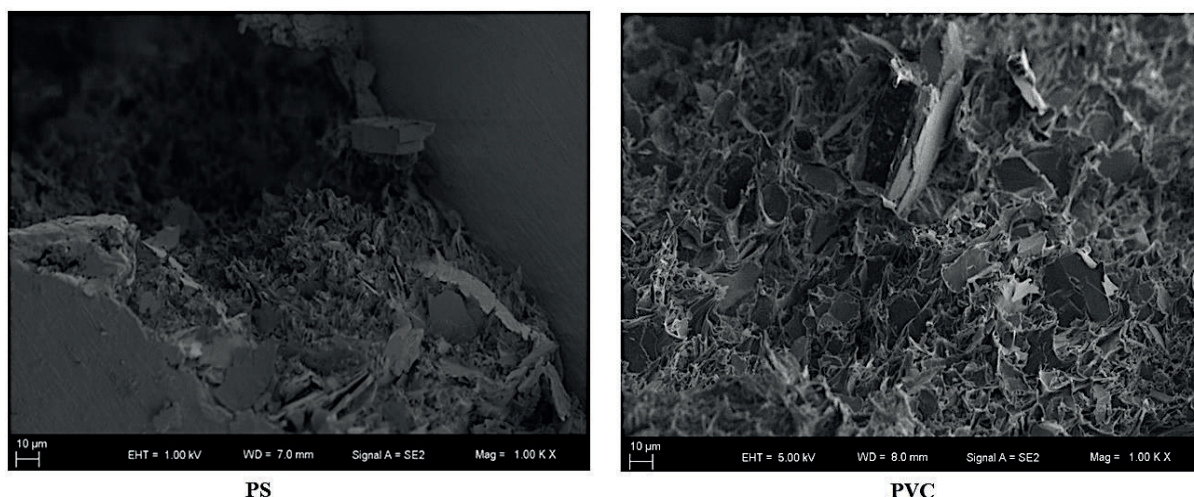


Figure 4. SEM analysis results for the prepared PS and PVC composite

3.5. Chemical analysis results

In this study, Fourier Transform Infrared (FT-IR) spectra of polyvinyl chloride (PVC), polystyrene (PS), zinc borate, and talc, which were used as raw materials in the experimental work, were recorded and analysed. Characteristic C–H stretching vibrations of polyvinyl chloride were observed in the FT-IR spectrum within the 2800–3000 cm^{-1} region. The bands appearing between 1300 and 1500 cm^{-1} indicated aliphatic C–H bending, and the absorption peak located at approximately 1250 cm^{-1} was related to C–H bending in close proximity to chlorine atoms. The formation of C–C stretching vibrations along the PVC backbone chain was identified by the peaks in the range of 1000–1100 cm^{-1} . Finally, the absorption bands observed between 600 and 700 cm^{-1} were assigned to the C–Cl gauche conformations.

Characteristic FT-IR bands of polystyrene included aromatic C–H stretching vibrations in the 2850–3100 cm^{-1} region. Aromatic C–H overtone and combination bands were identified between 1700 and 2100 cm^{-1} , while aromatic C–C stretching modes appeared in the 1450–1600 cm^{-1} range. Furthermore, deformation vibrations of aromatic C–H bonds were detected between 900 and 1200 cm^{-1} , with out-of-plane C–H deformation vibrations occurring in the 650–900 cm^{-1} interval. The FT-IR results obtained for polystyrene were consistent with those reported in the literature [30].

The FT-IR spectrum of zinc borate exhibited characteristic hydroxyl group absorption bands in the range of 3200–3450 cm^{-1} . The absorption bands between 650 and 1400 cm^{-1} were attributed

to characteristic boron–oxygen bonding vibrations. In addition, the peaks observed in the range of 900–1300 cm^{-1} were identified as the characteristic vibrations of zinc borate. The main absorption band corresponding to the $\text{ZnO} \cdot \text{B}_2\text{O}_3$ structural units was detected at approximately 3200 cm^{-1} .

For talc, FT-IR analysis revealed hydroxyl group vibrations associated with magnesium ($\text{Mg}-\text{OH}$) and silicon ($\text{Si}-\text{OH}$) at approximately 3650 and 3450 cm^{-1} , respectively. $\text{Si}-\text{O}-\text{Si}$ stretching vibrations were identified by a prominent absorption band near 1040 cm^{-1} , whereas a peak close to 650 cm^{-1} indicated the vibrational behaviour of the $\text{Si}-\text{O}-\text{Mg}$ bond (Figure 5).

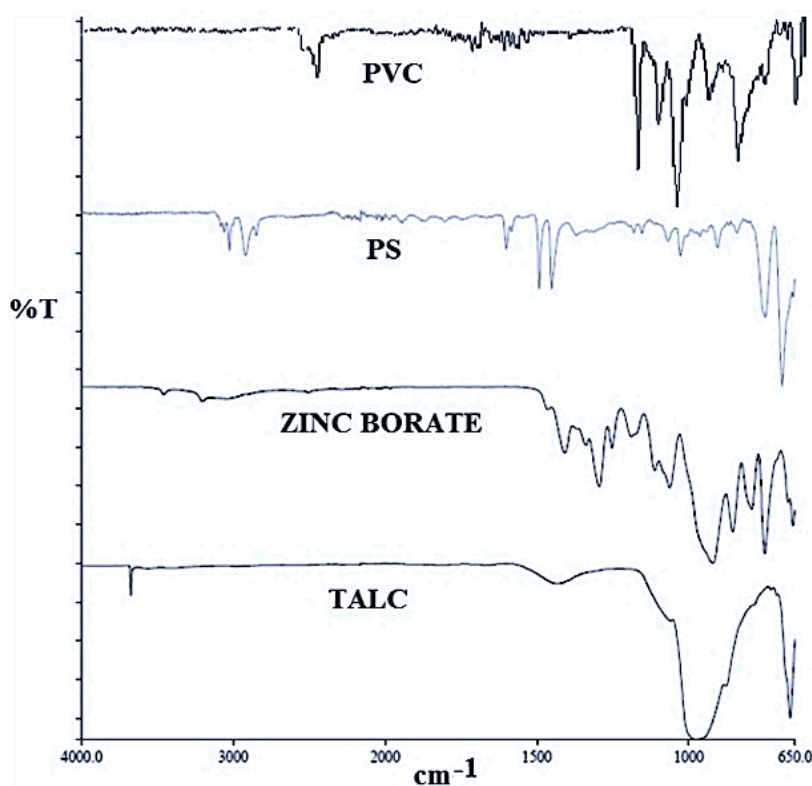


Figure 5. FT-IR analysis of raw materials

When compared with the FT-IR analysis of pure PVC, it was observed that there was no significant change in the chemical structure of PVC and the hydroxyl group in the structure was in the range of 3500–3800 cm^{-1} . When compared with pure PS, it is seen that there is no significant change in the chemical structure of PS, and the hydroxyl group in the structure is in the range of 3500–3800 cm^{-1} (Figure 6) [29].

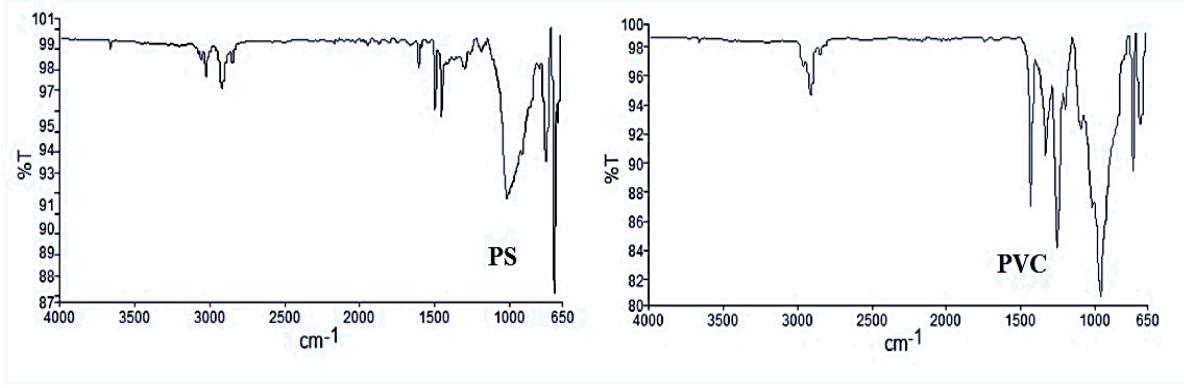


Figure 6. FT-IR analysis for the prepared PS and PVC composite

4. CONCLUSION

In this study, by adding various additives into plastic materials, the material was examined physically, chemically, and thermally. In the study, composite materials were prepared by adding zinc borate and talc into polystyrene, polyvinyl chloride. Polystyrene was dissolved in polyvinyl chloride dichloromethane and made ready for mixing with other additives. According to the weight of the polymer material, zinc borate and talc between 5-20%, temperature between 50°C-80°C and time between 5-20 minutes were determined. Impact strength test was performed on 32 prepared composites and according to the results, according to the Taguchi method, with the choice of 'The biggest is the best'; Based on the main effect values for S/N, the set-6 for PVC and the set-5 for PS were selected as optimum. Viscosity measurements were evaluated with FT-IR, TG/DTA, OIT, SEM analyses for the two selected sets. When the composites prepared in the FT-IR analyses were compared with the pure polymers, it was observed that there was no significant change in their chemical structures. When TG/DTA analyses were examined, it was observed that composite materials with zinc borate added to their structure decomposed at higher temperatures, less and slower than pure materials. According to the OIT analysis results, it was observed that the prepared PS samples were more resistant to fire than PVC. It was seen that the properties of polymer materials were improved with the study and evaluated in automotive sector.

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