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Research Article

Production and Characterization of Epoxy Resin-Based Biocomposites Reinforced with *Citrus maxima* Peel

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Abstract – In this study, epoxy resin-based biocomposite production and characterization with the addition of *Citrus maxima* (pomelo) peel is carried out. To increase the environmental sustainability of composites, pomelo peel is dried and ground as bio-waste and mixed into epoxy resin as a reinforcement material. During the experimental production process, optimization studies are carried out and different amounts of pomelo peel powder (0 wt.%, 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.%) are mixed homogeneously with the epoxy resin matrix and cast into standard molds. The mechanical, thermal, and morphological properties of the obtained biocomposites have been examined in detail. When the results are evaluated, both the bulk density and Shore D hardness of the biocomposite decrease with pomelo powder supplementation. When this type of biomass is added to the resulting epoxy-based biocomposite, the thermal conductivity coefficient also reduces. In the thermal decomposition experiments of the biocomposite, it is understood that pomelo supplementation increases thermal stability. When scanning electron microscope (SEM) images of the biocomposites have been investigated, it is seen that there is a good interfacial adhesion between the epoxy matrix and the pomelo peel. However, when Fourier-transform infrared spectroscopy (FTIR) spectra are examined, it is understood that this interaction is physical. These results show that pomelo peel-reinforced epoxy-based biocomposites can potentially be used as environmentally friendly and low carbon footprint biomaterials.

Keywords – Citrus Maxima, Biocomposite, Bulk Density, Hardness, Thermal Conductivity, Thermal Stability.

I. INTRODUCTION

Citrus, one of the most important fruits in the world, is a large family that includes sweet orange, tangerine or mandarin, grapefruit, lemon and lime. The history of *Citrus maxima* (Pomelo), which is included in this family, dates back to ancient times. This fruit, believed to have originated in Asia, has been cultivated and used for more than 4,000 years. It is an important species of cultivated citrus. China is one of the main production areas of pomelo and has a long history of cultivation for this product. There are more than 120 varieties of pomelo and pomelo has three production centers in China: South China region, Southeast coastal region and the Southwest and South regions. Today, it is cultivated in Asian countries as well as in America, Australia and other regions [1].

C. maxima, also known as shaddock, is one of the largest citrus fruits in terms of size. It derives its Latin name from this feature. This fruit is popular in Asia and Europe due to its desirable taste, flavor and juicy texture and is gaining popularity worldwide [2]. It is a perennial tree and edible fruit belonging to the Rutaceae family. Pomelo is a large fruit tree species belonging to the orange family. Originating in Asia, this fruit is cultivated in many Asian countries. Specific features that also contribute to the easy identification of the species include huge leaves on broad winged petioles, fragrant large-sized flowers. Among citrus fruits, pomelo fruits are morphologically considered the largest citrus fruit with a green color, round shape, a diameter of about 20 cm and an average weight of 1-2 kg. Due to its flavor and aroma, the endocarp (fleshy part of the fruit) of the pomelo fruit constitutes the edible part, while the fruit peel can be economically considered as agricultural waste [3-5]. The pomelo fruit is usually large and round in shape, with a yellowish green peel. The fleshy parts of the fruit are pink in color and have a sweet flavor. The fruit is always round in shape, large in size, and is a plant native to Asia, and is commercially grown in China and India [6].

Citrus peels are a good source of nutrients and phenolic compounds, and since they are produced in large quantities during fruit processing, these residues can be used as a potential resource instead of being disposed of as waste in the environment. Pomelo peel has many characteristics, like the fruit itself. *C. maxima* peel is thicker and harder than the peels of other citrus fruits. It usually has a smooth and shiny surface. This increases the attractiveness of the fruit and creates a visually pleasing effect when cooking or presenting. It can be greenish-yellow in color. However, it can also be brown, yellow or pinkish in color. Pomelo peels are also sweet and citrus-flavored and can be used in marmalade, candy and citrus-flavored dishes. Like other citrus species, they contain essential oils. These oils are responsible for the aroma of the peel and can also be used to give flavor and smell to foods. However, systematic studies on the determination of active substances and antioxidant capacity of flavedo and albedo of ripe fruits of local pomelo varieties are rare. Studies have shown that the health-promoting properties of pomelo are mainly due to flavonoids and phenolic acids, which have anti-inflammatory and anti-cancer properties that may play a role in the prevention of cardiovascular disease, diabetes and other diseases. One of the main mechanisms of the antioxidant effect of phenolics in functional foods is free radical scavenging. *C. maxima* and *Citrus sinensis* essential oils, essential oil combinations against fungi and aflatoxins were found to be significantly effective against fungal growth in vitro. Pomelo, which contains high amounts of vitamin C, is beneficial for immune system health. In addition, it protects the body against diseases due to its richness in antioxidants and phytochemicals. It has been recommended in traditional herbal medicine as a source of diabetes medicine or antidiabetic medicine. *Citrus* fruits contain flavonoids and limonoids, which have been proven to have anti-inflammatory and antitumor activities. *C. maxima* leaves are known to contain antihyperglycemic and antioxidant properties [6-10].

Different parts of *citrus* peels have potential anti-inflammatory properties due to their phenolic and flavonoid contents. The main phenolic compound found in the mesocarp and exocarp of citrus peels was found to be hesperidin. Different amounts of phenolic compounds such as hesperidin, t-ferulic acid, catechin, sinapic acid, vanillin, chlorogenic acid and caffeic acid were detected in *C. maxima* peel extracts. One of the main mechanisms of antioxidant effect of phenolics in functional foods is free radical scavenging. Studies have shown that the basis of the resistance property of *C. maxima* is the rich antioxidant source of its outer peel and inner peel (albedo) [11-21].

Citrus fruits produce a lot of waste and if the waste is not managed properly, it starts to rot and smell. When decay begins, the biochemical reactions that occur endanger life in the environment. Approximately 2 million tons of citrus waste are produced every year. A large part of fruit waste (up to 90%) such as citric acid, essential oils, limonene, dry pulp, pectin can be converted into usable products. The usability of these wastes in obtaining high value-added products is gaining popularity day by day. It is also likely to be used as animal feed. The peel has a very thick foamy part. The peel part constitutes 30% of the pomelo fruit by weight. The thickness of the peel can vary between 1.25-2 cm. Pomelo peel foam can be considered a great source for cellulose extraction. The pomelo peel has a foam layer that has the ability to dissipate kinetic energy. When the fruit falls from the tree, this foam shell absorbs the impact of the ground and prevents damage to the pulp, thus acting as a damping or shock absorbing structure. Sodium and potassium are the two most important ions that are key players in maintaining the electrolyte balance between the cellular and extracellular environment and among other fluids present in a living system. It has been reported that *citrus* fruits contain more potassium than sodium. The peel of *C. maxima* was found to contain 7.5 times more sodium than its pulp. Furthermore, the potassium concentration in the peel is 20% higher than the potassium value in the pulp [23-28].

Due to increasing environmental concerns, interest in green composites is increasing. Natural and ecologically safe products are preferred. The use of natural materials as additives in thermoplastic materials is a developing interest in the field of polymer composites. The use of natural materials in a synthetic polymer matrix to make natural polymers is attracting attention. Natural polymers tend to be more susceptible to degradation due to the inclusion of natural materials as fillers/additives/reinforcements. They are used in various fields such as packaging, controlled drug delivery, wound dressing, food, engineering. Pomelo peel has recently been investigated as an alternative material in the production of composite materials. The peel contains fibers that can be used as natural fibers and therefore can be used in the production of bio-composite materials by combining with polymer matrices. The use of such composite materials is seen as an environmentally friendly and sustainable option that can replace traditional synthetic materials. It is possible to see *C. maxima* as an additive in such composite studies [29-32]. In addition, studies have also been conducted where pomelo peels are used as materials in the production of various nanoparticles as green synthesis. Due to the high waste rate of pomelo, it has been used as ‘reducing and capping’ agents in the synthesis of green nanoparticles within the economic and environmental model. Due to its sensitivity to iron deficiency, it is also preferred in the production of iron oxide nanoparticles through green synthesis [33-36].

In recent years, there has been a growing interest in developing sustainable biocomposites that incorporate natural and waste-derived materials, driven by the need to reduce environmental impact and reliance on non-renewable resources. Organic fillers, such as agricultural and food industry by-products, have emerged as promising reinforcements due to their biodegradability, cost-effectiveness, and ability to enhance certain material properties [37-39]. *Citrus maxima* (pomelo) peel, a common food waste, is particularly notable for its potential as a reinforcing agent, given its fibrous structure and availability. This study investigates the incorporation of pomelo peel powder into an epoxy resin matrix to produce a biocomposite with improved environmental sustainability. Through systematic optimization, different filler ratios are tested to evaluate their effects on the mechanical, thermal, and morphological properties of the resulting biocomposites. By determining the optimal filler concentration, this research aims to contribute valuable insights into the development of lightweight, thermally insulating, and eco-friendly materials that utilize pomelo peel as an effective bio-waste reinforcement.

II. MATERIAL AND METHOD

In this study, the production and characterization of biocomposites based on epoxy resin reinforced with *Citrus maxima* (pomelo) peel were carried out. For the preparation of the composites, firstly *Citrus maxima* peels were reduced to certain particle sizes by drying and grinding processes. The obtained particles were added to the epoxy resin matrix in certain ratios. As epoxy resin, commonly used bisphenol-A based epoxy resin (EPON 828) and a suitable hardener (polyamine based) were used. These materials were precisely weighed and mixed in certain ratios and mixed thoroughly with a magnetic stirrer and ultrasonic mixer in order to obtain a homogeneous distribution [40-43]. After the mixing process was completed, the composite mixture was poured into molds and left to cure under control at 50 °C. The curing process ensured that the epoxy resin reached maximum strength and structural integrity. The necessary physical tests, chemical analyzes and characterization processes were performed for the samples obtained after curing. Surface morphology, bulk density, Shore D hardness, and thermal conductivity coefficient were determined [44-47]. Figure 1 shows the fruit, peel, and ground powder form of the organic filler (*Citrus maxima*) used in the studies. In addition, in this study, 1 wt.% modified castor oil was added to the epoxy-based biocomposite in order to improve its plasticizing properties. Thus, the low temperature performance and workability of the composite were increased. In addition, modified castor oil and organic fillers were physically hydrogen bonded [48, 49]. The mixing ratio of Epoxy A and Epoxy B was taken as 2/1 by mass.

Figure 1. *Citrus maxima* (pomelo) peel and ground form used in experimental studies

Figure 2 shows the biocomposite production scheme using organic filler. In the first stage, it is very important for Epoxy A and the filler to form a homogeneous mixture. In the second stage, Epoxy B is added and attention is paid to the curing time at optimum temperature and time.

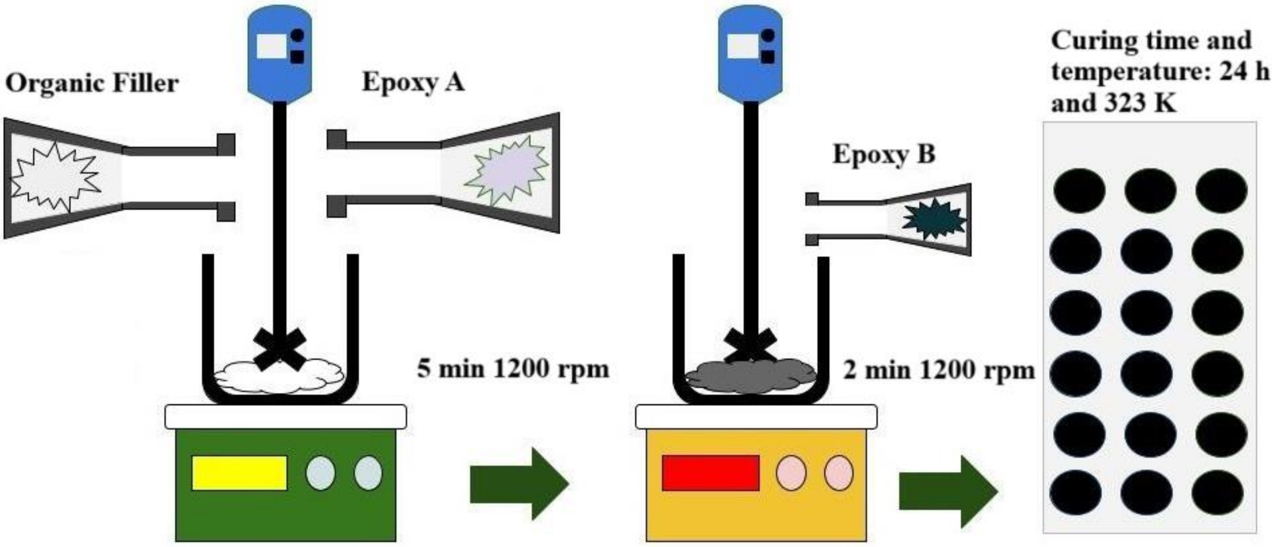

Figure 2. Production scheme of organic filler reinforced epoxy based biocomposite

III. RESULTS AND DISCUSSION

Physical Test and Chemical Analysis Results of Epoxy Based Biocomposite

In this study, a comprehensive overview of the properties of the epoxy-based biocomposite with organic filler addition is presented. *Citrus maxima* (pomelo) shell filler added to the epoxy matrix reduces the bulk density of the biocomposite and provides lightness. This makes the composite material lighter, providing ease of transportation, energy efficiency and use in various applications. The volumetric density-reducing effect of the filler is due to the increase in pores in the structure of the composite and the lower density of the organic filler. Accordingly, a decrease in Shore D hardness is also observed. This decrease in hardness becomes more pronounced with the increase in the amount of filler and can also affect the flexibility properties of the composite [50]. Although this decrease in hardness leads to limited use of the biocomposite in applications requiring durability, it offers an advantage for areas requiring low-density and flexible materials. In addition, a decrease in the thermal conductivity coefficient of the biocomposite was observed with the addition of the pomelo shell filler. This decrease in thermal conductivity shows that the biocomposite can be used in areas requiring thermal insulation and is especially important in terms of energy saving.

The thermal stability of the biocomposite varies depending on the filler ratio. While a good interface fit is provided between the filler and the epoxy matrix at low filler ratios, it has been understood that the filler ratio should be optimized around 3 wt.%. At the optimum filler ratio, thermal stability remains at a relatively high level and the positive effect of the filler on the composite is observed. However, when the filler ratio exceeds 3%, increased porosity and rough surface morphology increase the brittleness of the biocomposite and negatively affect thermal stability. According to the thermal analysis results, the heat resistance of the biocomposite decreases at higher filler ratios, which is a limiting factor in applications requiring structural integrity under high temperatures. In terms of surface morphology, SEM images show that the physical interaction between the biocomposite matrix and the pomelo peel provides a good interface fit. However, it was observed that this fit deteriorates at high filler ratios and the composite structure becomes porous. These results indicate that epoxy-based biocomposites can be used as environmentally friendly and low carbon footprint materials at optimal filler ratios, but they also emphasize that high filler ratios should be carefully balanced [51]. In Figure 3, it is understood that the bulk density of the epoxy-based biocomposite decreases as the organic filler ratio increases. Figure 4 shows that the addition of organic filler reduces the Shore D hardness of the biocomposite.

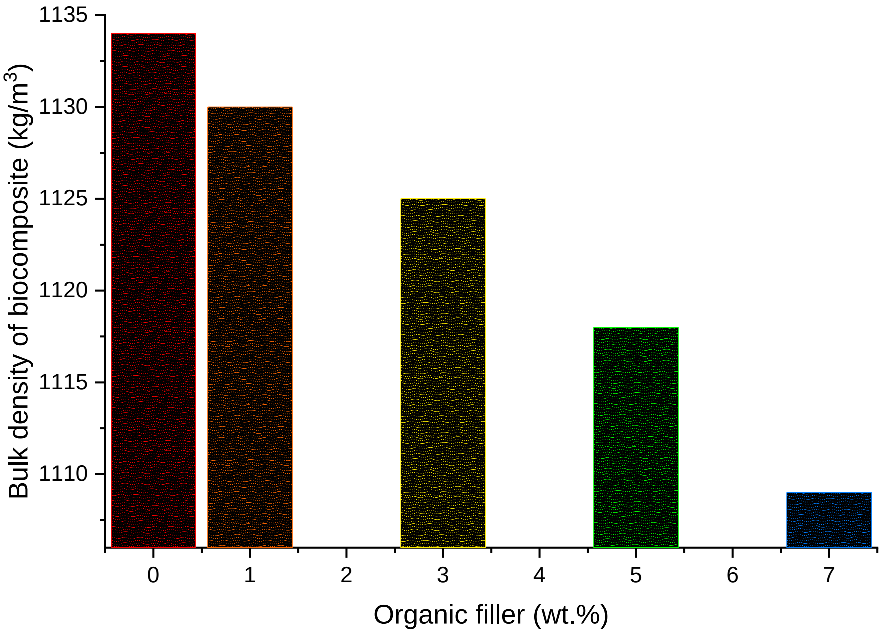

Figure 3. Effect of organic filler on bulk density of epoxy based biocomposite

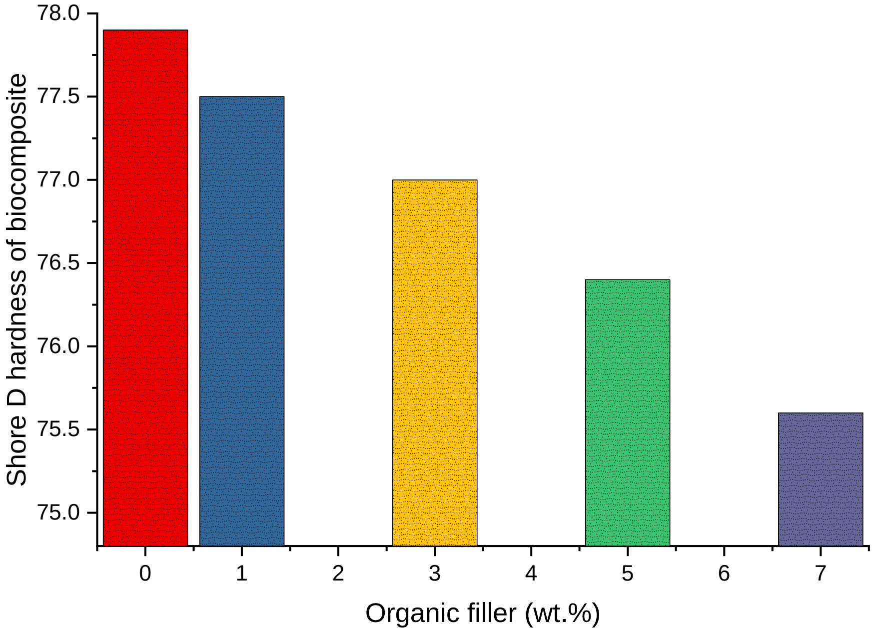

Figure 4. Effect of organic filler on Shore D hardness of epoxy based biocomposite

The addition of *Citrus maxima* peel to the epoxy matrix improves the thermal insulation properties by reducing the thermal conductivity coefficient of the biocomposite. This decrease in the thermal conductivity coefficient is due to the structural properties of the filler. Pomelo peel has a low thermal conductivity value due to its organic structure, and the porous structure created by the biocomposite in the matrix structure partially interrupts the heat transfer paths and provides a lower thermal conductivity. Figure 5 shows that the organic filler reduces the thermal conductivity coefficient of the epoxy-based biocomposite. This feature enables the use of the biocomposite in areas where thermal insulation is required, contributes to energy saving and makes it suitable for environmentally friendly applications. This effect of the addition of organic filler offers significant advantages, especially in building materials with low density and insulation properties. However, if the filler ratio exceeds the optimal level, the decrease in thermal stability stands out as a factor that should be considered in applications requiring heat resistance. Therefore, the correct adjustment of the organic filler ratio is of critical importance to optimize the heat transfer performance of the biocomposite [52].

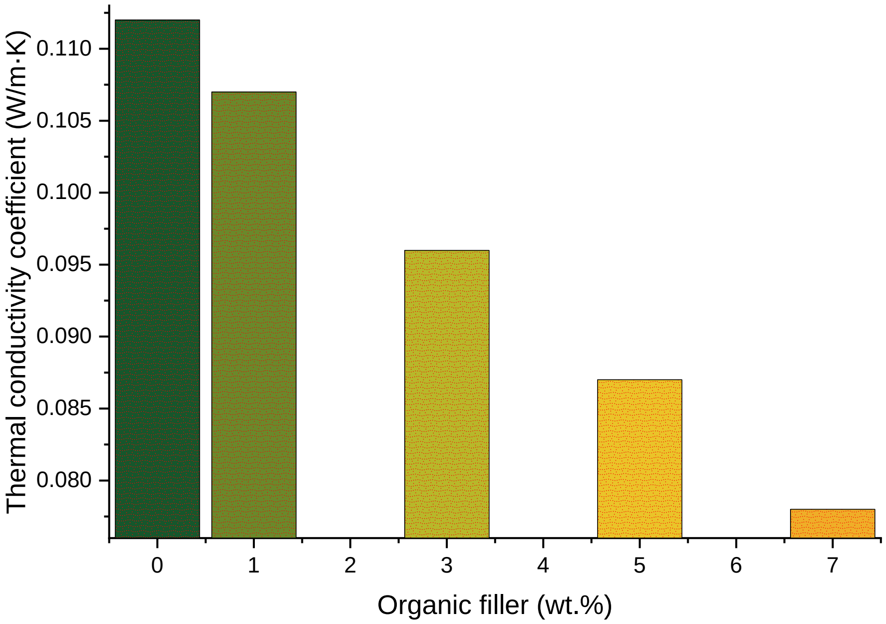

Figure 5. Effect of organic filler on thermal conductivity coefficient of epoxy based biocomposite

Fourier transform infrared spectroscopy (FTIR) analysis of epoxy-based biocomposite containing *Citrus maxima* peel (3 wt.%) provides valuable information about the structural properties of the composite and the interactions between the filler and the epoxy matrix (Figure 6). When the FTIR spectra were examined, it was observed that the characteristic peaks belonging to the epoxy resin were evident, but the addition of the pomelo peel filler did not cause a chemical change in these peaks. The C–O–C stretching vibration specific to the epoxy matrix was observed as a strong peak around 1100 cm^{-1}, while the aromatic C=C stretching vibrations were observed in the range of 1500-1600 cm^{-1}. These bands were largely preserved when the filler was added, indicating that there was no chemical change in the basic structure of the matrix. This indicated that there was a physical interaction between the epoxy and the pomelo peel and that no chemical bonding occurred. It is observed that OH stretching vibrations increase slightly in the range of 3300-3500 cm^{-1} with the contribution of organic filler, which is due to hydroxyl groups naturally found in pomelo peel. These bands indicate that the hydrophilic groups on the surface of the filler are physically bonded to the epoxy matrix and do not cause a chemical change in the matrix. In addition, slight changes are observed in the range of 2800-3000 cm^{-1} regarding C–H stretching vibrations, which indicate that the changes in surface energy are physically reflected in the matrix when the filler is added [53]. No significant chemical reaction or new functional group formation is observed in the FTIR spectra at 3 wt.% pomelo filler ratio, confirming that the filler in the biocomposite is physically incorporated into the epoxy matrix. These results indicate that despite the increase in the filler ratio, the epoxy matrix structure maintains its chemical integrity and the filler acts as a physical reinforcement in the composite. This type of interaction can be explained as the source of the observed changes in the mechanical and thermal properties of the biocomposite and reveals that the organic filler acts as a physical reinforcement to achieve optimal performance in epoxy-based biocomposites.

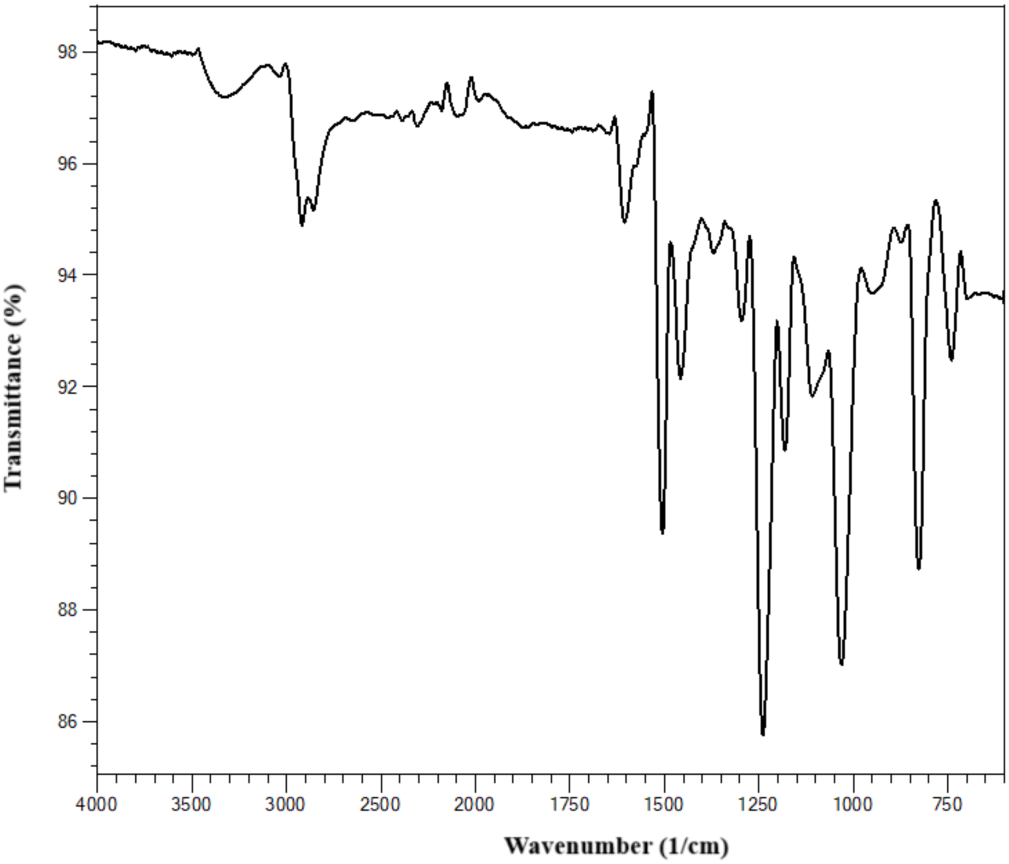

Figure 6. FTIR spectra of epoxy-based biocomposite with 3 wt.% organic filler addition

The surface morphology of epoxy-based biocomposites to which *Citrus maxima* peel was added at 0 wt.%, 1 wt.%, 3 wt.%, 5 wt.% and 7 wt.% was analyzed in detail with scanning electron microscope (SEM) images. While the surface of the undoped epoxy matrix exhibited a homogeneous and smooth structure, there was no observable void or porosity on the surface. This pure epoxy structure presents a completely dense and tight surface morphology without any organic filler effect. With the addition of 1 wt.% pomelo peel, small-diameter, homogeneously distributed filler particles began to be observed in the matrix. The small and well-distributed filler particles indicate that the interface between the matrix and the filler is well-matched and that no serious voids are formed. This ratio allows the biocomposite to maintain its surface integrity to a large extent. The addition of 3 wt.% filler was determined as the optimal filler amount, and a compatible interface between the matrix and the filler was observed in the SEM images (Figure 7). The homogeneous distribution of filler particles at this ratio indicates that the surface morphology is stable and the filler is well-integrated with the epoxy matrix. This compatible structure increases the mechanical strength of the biocomposite while ensuring that the porous structure observed on the surface remains at a minimum. At a 5 wt.% filler ratio, a slight porosity was observed on the surface as the density of the filler particles increased (Figure 8). These pores are due to the filler not being fully integrated with the epoxy matrix, leading to the formation of microscopic voids between the filler particles. This situation causes irregularities and slight roughness on the surface, indicating a deterioration in the surface morphology of the composite. At a 7 wt.% filler ratio, these effects become more pronounced. In SEM images, it is seen that the filler particles are clustered and the interface compatibility between the matrix and the filler decreases. At this high filler ratio, larger voids and irregular distributions occur between the filler particles. This situation leads to more pronounced porosity and roughness on the surface, creating a surface structure that may negatively affect the mechanical strength of the composite [54]. With the increase in the filler ratio, the absence of sufficient epoxy matrix between the filler particles causes the matrix-filler bond to weaken and the fragility of the biocomposite to increase. Therefore, a 3 wt.% filler ratio provides an optimum structure in terms of surface morphology; at higher ratios, surface integrity is impaired and composite performance decreases. These analyses show that the correct selection of the filler ratio in epoxy-based biocomposites has a critical effect on the surface morphology and therefore the overall performance of the composite material.

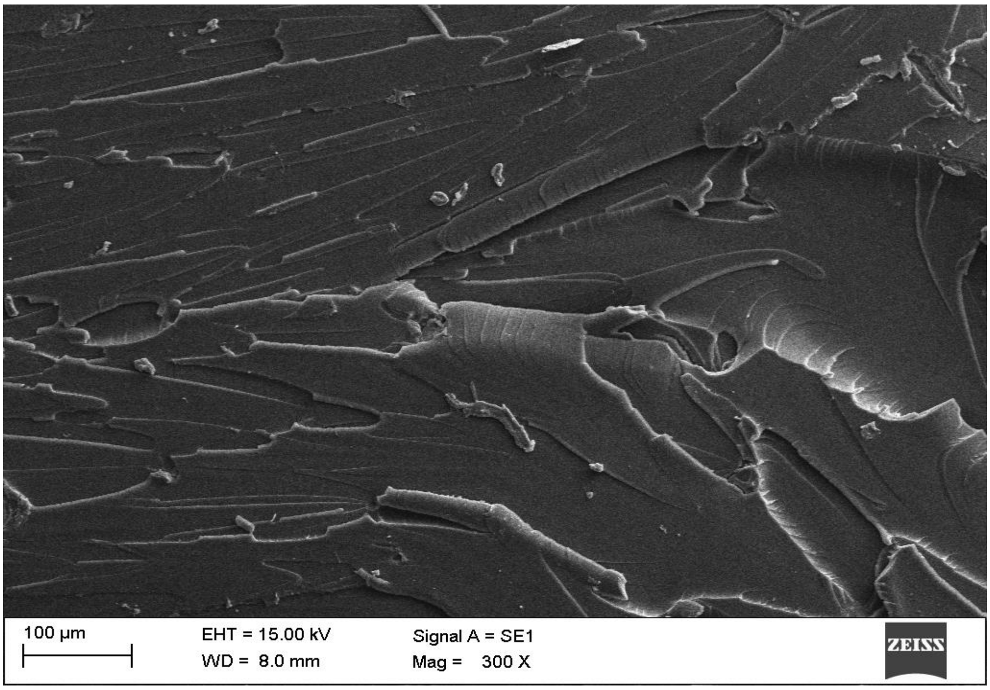

Figure 7. SEM image of epoxy-based biocomposite with 3 wt.% organic filler addition

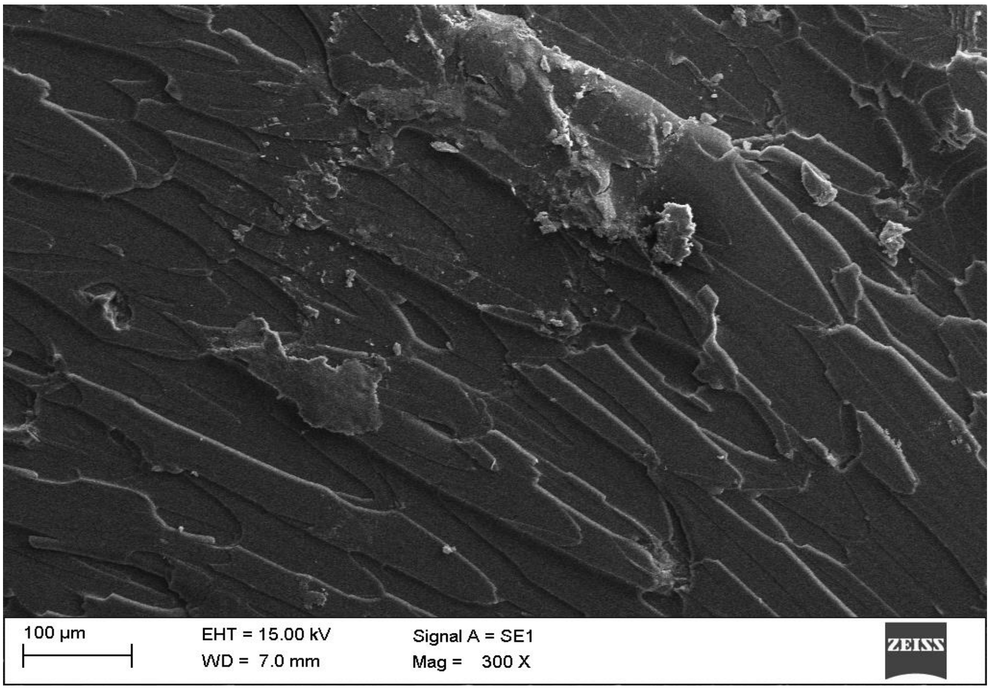

Figure 8. SEM image of epoxy-based biocomposite with 5 wt.% organic filler addition

IV. CONCLUSIONS

This study reveals that the incorporation of *Citrus maxima* (pomelo) peel as an organic filler in epoxy-based biocomposites effectively reduces the bulk density, Shore D hardness, and thermal conductivity coefficient of the material, making it lighter and potentially more suitable for thermal insulation applications. The addition of pomelo peel also decreases the biocomposite's thermal stability, as observed in thermal decomposition tests. SEM analysis further demonstrates good interfacial adhesion between the epoxy resin and pomelo peel particles at optimal filler levels, contributing to a uniform morphology and supporting the physical integrity of the composite. However, as the filler content increases, the Shore D hardness decreases, pointing to a softening effect imparted by the organic reinforcement. FTIR analysis confirms that the interaction between the epoxy matrix and pomelo peel is primarily physical, emphasizing the need for optimal filler ratios to maintain desired mechanical properties. According to the results obtained, 3% organic filler reinforced biocomposite was determined as the optimum ratio. It was determined that the mechanical properties, surface morphology and brittleness of the composite increased at higher ratios. These findings underscore the potential of pomelo peel waste as a sustainable reinforcement material in biocomposites, offering a pathway to environmentally friendly and low-carbon footprint biomaterials with tunable properties for various applications.

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