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Mechanical and tribological properties of epoxy composites reinforced with food-waste fillers

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ABSTRACT

Purpose: One of the assumptions of the zero-waste economy is to reduce the amount of industrial waste produced, process it, and recover it without burning or burying it. Citrus peels are among the food wastes that are difficult to recycle. Due to the long time of decomposition and the waxes and fats in their structure, the shells rarely end up in composters and, consequently, are not included in natural fertilisers. The assumptions fit into the research described in the article.

Design/methodology/approach: The authors investigated the possibility of using ground peels of citrus fruits: grapefruit, key lime, lemon and orange as fillers in composite materials in which the role of the matrix was played by epoxy resin. Composite materials with 2.5, 5 and 10% filler content were prepared. The materials were tested using the tensile, hardness, and abrasive tests using the pin-on-disc method.

Findings: The research was to answer whether adding citrus waste can change the physicochemical properties of composite materials based on epoxy resin and native resin. Particular attention was paid to the properties that are important from the point of view of engineering applications: mechanical properties and tribological properties.

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Practical implications: In the face of challenges related to the growing amount of waste from the food industry, joint materials engineering tries to answer whether this waste can be used in the production of composite materials. In several publications from recent years, it has been postulated that used food industry products can be used as fillers for composite materials, as they can, on the one hand, improve specific physicochemical properties of new materials and manage food waste.

Originality/value: The tests proved that composite materials with grapefruit and key lime as a filler were characterized by the best tribological properties, mechanical properties, and hardness, which were unchanged or better than the epoxy resin used as a matrix.

Keywords: Composites, Mechanical properties, Abrasion, Epoxy resin, Natural filler

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PROPERTIES

1. Introduction

The modern industry is constantly looking for construction materials that can replace the engineering materials used due to their improved functional properties and increased durability [1,2]. Continuous progress in materials engineering makes it possible to manufacture new or improved construction materials, which must undergo extensive testing before their production can be allowed [3,4]. Much interest in the manufacturing industry is currently focused on using composite materials. Composite materials have been used for a long time due to their good construction properties, low specific weight, variety of processing techniques, and the possibility of differentiating properties by modifying intermediate inputs and processing techniques [5,6]. In addition, their use was popularised because it was possible to introduce changes in structural properties by including modifiers in their composition. Physical modifiers and reinforcing materials improve material properties such as density, hardness, impact strength, mechanical and tribological properties, and thermal stability [7-13]. The newly developed materials and their possible degradation products must also be non-toxic to normal cells in the human body [14]. The research was carried out to determine the influence of various physical modifiers on the physicochemical properties of polymer composites. The popular modifiers include inorganic compounds such as: $CaCO_3$, CuO, ZnO, SiC, ZrO_2, Si_3N_4, TiO_2, $CaSiO_3$, SiO_2 and Al_2O_3 [15-17]. The aspects related to the composition of composites concern not only the modifiers themselves but also the reinforcement in the form of fabrics. Using various plastics as a matrix gives the possibility of modifying the properties of the composite [18,19].

Industrially, polymer composites reinforced with synthetic fibres (e.g., glass, aramid, ceramic) play the most significant role and are used in many industries, such as aerospace, self-driving cars, and construction [20]. Composites reinforced with organic fibres play an ever-increasingly important role. They are characterised by good strength, lightness, composite stiffness, low cost, low friction coefficient, adequate resistance to abrasive and erosive wear, and, most importantly, biodegradability (including lower disposal costs) [21]. It aligns with growing environmental awareness and concepts of sustainability (responsible waste management and product design with recycling in mind) [22].

Various waste materials in agriculture, livestock farming, or the food industry produce suitable biocomposite fillers. Several papers report using biological materials like plant parts [23]. Examples of such applications include walnut shells, lask nut shells, and sunflower seed shells to improve the stiffness of epoxy resins [24]. Orange processing waste has been used as a filler to enhance mechanical properties [25] and sliding wear resistance [26]. Pineapple leaves [27] and peanut shells [28] have been used for insulation.

So far, Hitesh Sharma and his team researched using citrus limetta peel (CLP) as fillers in composite materials. In a 2019 publication, they examined the effect of citrus limetta peel (CLP) on the mechanical properties of epoxy resin composite materials [25]. A 2020 study examined the influence of incorporating citrus limetta peel (CLP) fillers at diverse loadings (5%, 10% and 15%) on the dynamic mechanical properties of epoxy composites [29]. Subsequent studies investigated the influence of CLP filler size on the thermal and mechanical behaviour of epoxy resin-based composites [30]. In another study, the influence of epoxy resin matrix composites with 15% CLP content on the wear behaviour of epoxy composites has been experimentally explored [31]. Research from 2023 aimed to study the potential of citrus limetta peel (fruit waste) as a novel filler in thermoplastic composites [32]. Poikelispää et al., polyacrylate rubber's dielectric and actuation properties (ACM) were studied after adding different vegetable-based fillers such as potato starch, corn starch, garlic, and paprika [33]. Mrówka et al. investigated the effect of using organic waste from citrus peels, including grapefruit, lime, lemon, and orange peels, on silicone-based composites' physical properties [34].

The paper aims to test the effect of citrus residues (grapefruits, key lime, lemon, and orange peels) as fillers for silicone-based composites. The study aims to determine how a given waste and its content affect the operational properties of the material (static tensile test and resistance to abrasive wear).

2. Materials and methods

2.1. Materials

The fillers used in this research were waste citrus peels, which the authors generated. The peels of the grapefruit (*Citrus paradisi*), key lime (*Citrus aurantifolia*), lemon (*Citrus limon*), and orange (*Citrus sinensis*) were used. Epidian 601 epoxy resin with TFF hardener (Ciech, Nowa Sarzyna, Poland) was used for the tests.

2.2. Fillers characterisation

The grain composition analysis was performed using a laboratory screener Fritsch ANALYSETTE (Fritsch, Idar-Oberstein, Germany) equipped with a set of sieves with mesh sizes of 0.32 mm, 0.22 mm, 0.18 mm, 0.125 mm, 0.065 mm, 0.03mm (Fritsch, Idar-Oberstein, Germany). The duration of the sieve analysis for each of the tested powders was 60 minutes.

Microscopic analysis was performed using a SUPRA 35 high-resolution scanning electron microscope (Carl Zeiss AG, Birkerod, Germany). Before microscopic examination, the powders were sputtered onto the carbon strip using an SCD050 BAL-TEC sputter (Capovani Brothers Inc., Scotia, New York).

2.3. Composites preparation

Before introducing the fillers into the epoxy resin, they were dried in a laboratory oven at 100°C for 3 hours until a constant weight was obtained. The epoxy resin was mixed with the hardener in a proportion of 100:27 by weight, and then the filler was added. The silicone components were mixed with the fillers on a high-shear mixer with a mixing speed of 500 rpm. In such a way composites were produced with a filler content of 2.5, 5 and 10% by weight. The materials produced are presented in Table 1.

Table 1.
Composition of samples

Material	Filler	Filler content, %
REF	-	0
2.5G	grapefruit	2.5
5G	grapefruit	5
10G	grapefruit	10
2.5K	key lime	2.5
5K	keylime	5
10K	keylime	10
2.5L	lemon	2.5
5L	lemon	5
10L	lemon	10
2.5O	orange	2.5
5O	orange	5
10O	orange	10

Test samples were cast by gravity casting into silicone moulds. The samples were allowed to cure for 72 hours. After this, the samples were subjected to the tensile and abrasion tests using the pin-on-disc method. All tests of composite materials were carried out at 20°C and 60% humidity.

2.4. Composites characterisation

Mechanical tests were carried out by EN ISO 527-1 for five samples from each test group. The tests were conducted on the MTS Insight 2 testing machine equipped with an extensometer. The stretching speed of the samples was 50 mm/min. During the tensile test, peak load, peak stress and Young's modulus were measured. The results were presented as the arithmetic mean of 5 measurements and the standard deviation.

The abrasion test was performed on the Tribometer CSM Instruments (Needham, Massachusetts, USA) by ASTM G99. The samples for testing were in the shape of cylinders with a height of 10 mm and a diameter of 12 mm. Before the test, the samples were cleaned with technical ethanol. The ball moving after the sample, with a dimension of 6 mm, was made of zirconium dioxide. The ball was pressed against the sample with a force of 20 N. The ball's linear speed was 10 cm/s. The abrasion for the tested materials was defined as a change in the coefficient of friction (μ) on the 20 m road.

For each tested group of materials, three measurements were made, for which the arithmetic mean was given.

Measurements of abrasive wear on the surfaces of the analysed samples were carried out using the SUTRONIC 3+ profilographometer (Taylor Hobson Inc., Warrenville, USA) and the dedicated TalyProfile Lite software (version 6.1.6101). All tests were conducted at room temperature (22°C, humidity: 50%).

3. Results

3.1. Fillers characterisation

The grain size analysis of crushed grapefruit, key lime, lemon and orange peels is shown in Figure 1. A remarkable similarity in the grain composition of grapefruit and lemon was noted, for which more than 85% wt. of all grains ranged from 0.125 mm to 0.18 mm. However, in the case of grapefruit peels, nearly 45% wt. grains remained on the sieve with a mesh size of 0.18 mm, and it was about 50% wt for lemon peels. A similar relationship can be seen in the grain size of limes and oranges, for which 68.4% wt. and 77.7%. The grains were in the grain class 0.3-0.125 mm; in both cases, about one-third were 0.065-0.125 mm. Given the above, it can be concluded that the grain composition of the crushed lime and orange peels contained a more significant number of grains smaller than 0.125 mm.

In the microscopic photos (Fig. 2) of the analysed crushed citrus peels, it can be seen that the shapes of the seeds were almost the same for all citrus peels, and the only distinguishing feature of the seeds was their size. Irregular and globular grains (mainly for grains over 75 µm) and flake-shaped grains (especially grains below 10 µm) occurred here. It should be noted that the peel grains of the analysed citruses had a characteristic layered structure (Fig. 2). Therefore, their deeper grinding will cause their cracking (detachment) along the cleavage plane and thus cause the formation of more flake grains.

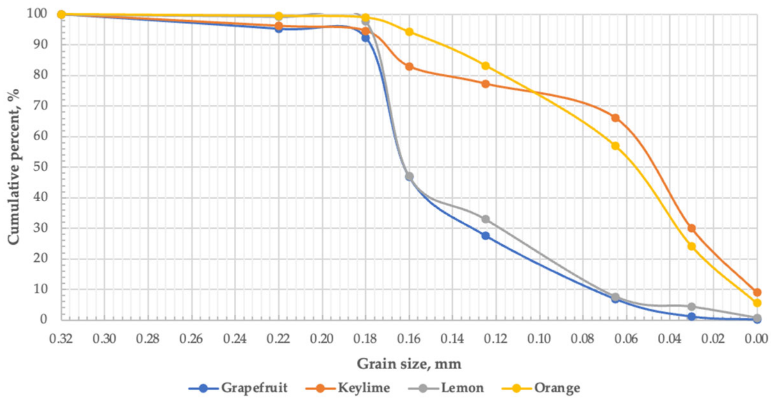

Fig. 1. Particle size distribution

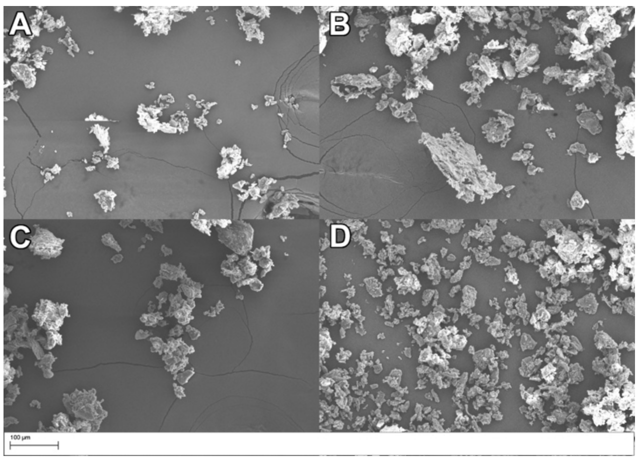

Fig. 2. SEM analysis of the powders: A – Grapefruit, B – Keylime, C – Lemon, D – Orange

3.2. Composites characterisation

The tensile test results are shown in Figure 3 (peak load), Figure 4 (peak stress) and Figure 5 (Young modulus).

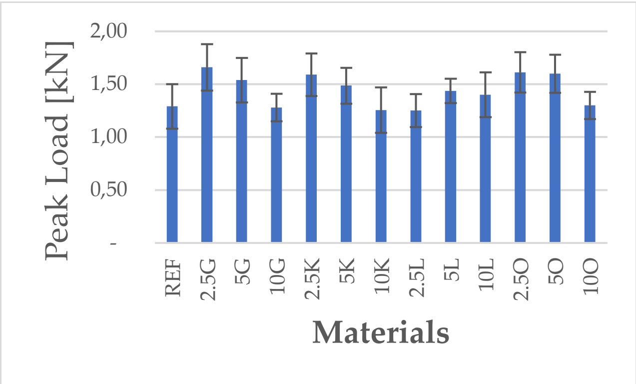

Fig. 3. Peak load of tested materials

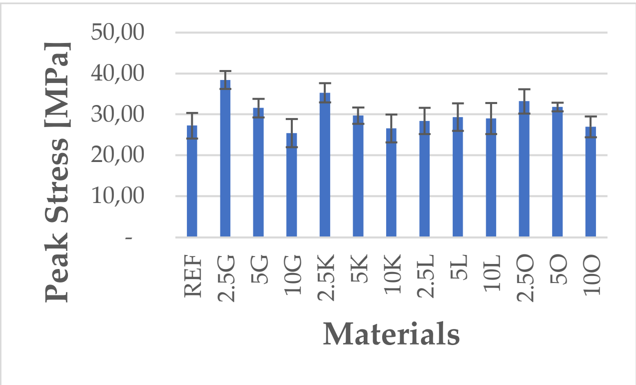

Fig. 4. Peak stress of tested materials

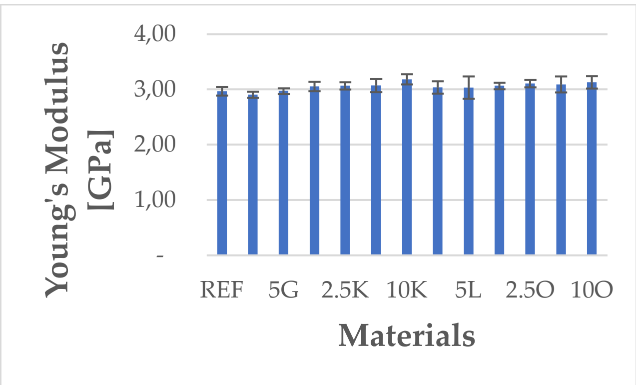

Fig. 5. Young's modulus of tested materials

The peak load value for epoxy resin without adding fillers is 1.29 kN. For composite materials, the filler was grapefruit. It was noticed that as the filler content increases, the peak load value decreases. The peak load for 2.5G material is 1.66 kN, for 5G material 1.54 kN, and for 10G material 1.28 kN. The peak load value for the 2.5G material was the highest among all tested materials and was 29% higher than the peak load value for the resin without adding filler. A similar tendency was observed for materials with key lime was the filler. The filler was grapefruit. Also observed here is that the peak load value decreases as the filler increases. For the material in which the key lime content was 2.5%, the peak load value was 1.59 kN; for the 5K material, it was 1.49 kN; and for the 10K material, it was 1.26 kN. Composite materials showed a different tendency, where the filler was lemon. The lowest value characterised the material with the lowest filler content (2.5L). The peak load value for the 2.5L material was 1.25 kN, which was the lowest peak load value measured for all tested materials and was lower than the peak load value for the reference material by 3%. Peak load values for composite materials containing 5% and 10% lemon filler amounted to 1.44 kN and 1.40 kN, respectively. For materials. where orange was the filler, the peak load values for 2.5O and 5O materials are 1.61 kN and 1.60 kN, respectively. The values should be considered the same for both materials. For the 10O material, a decrease in the peak load value to 1.30 kN was recorded.

The peak stress value for the REF material was 27.26 MPa. For composite materials containing grapefruit as fillers. key lime and orange, a concurrent trend was noticed. With the increase in the filler content, the peak stress value in these groups of tested composite materials decreases. For composite materials in which the filler is grapefruit, the peak stress value is 2.5G – 38.42 MPa, respectively. 5G – 31.58 MPa and 10G – 25.44 MPa. The peak stress value for the 2.5G material was 38.42 MPa, which was the highest value among the tested materials and was higher than the peak stress value for the reference material by 41%. The stress peak value for the 10G material was 25.44 MPa, the lowest measured value among all tested materials, and it was lower than the peak stress value for the REF material by 7%. For composite materials, in which the key lime is the filler, the peak stress value is 2.5K – 35.30 MPa, respectively. 5K – 29.70 MPa and 10K – 26.58 MPa. The peak stress value for composite materials with orange filler is 2.5O – 33.20 MPa, respectively. 5O – 31.86 MPa and 10O – 27.00 MPa. Composite materials showed a different tendency when the filler was lemon. The peak stress value for materials with this filler is 28.40 MPa (2.5L), respectively. 29.36 (5L) and 29.02 MPa (10L). The values should be considered close to each other.

Young's modulus value for the resin without adding fillers was 2.96 GPa. For composite materials with filling in the form of grapefruit, a slight increase in Young's modulus value was observed with the increase in the filler content. Young's modulus for the 2.5G material was 2.90 GPa, and for the 5G material, it was 2.97 GPa. Meanwhile, for the 10G material, it is 3.05 GPa. The measured value for the 2.5G material was 2.90 GPa, the lowest of the measured values and was 2% of the value calculated for the unadded resin. Young's modulus values for 2.5K and 5K were similar for composite materials with key lime filler and amounted to 3.06 and 3.07 GPa, respectively. Young's modulus value for the 10K material was 3.18 GPa. It was the highest Young's modulus value of all materials tested and was 7% higher than Young's modulus value measured for the REF material. Young's modulus values for composite materials with lemon filling were similar and amounted to 3.04 GPa (2.5L), 3.03 GPa (5L) and 3.06 (10L), respectively. A similar tendency was observed for materials with orange filler – 3.10 GPa (2.5O), 3.09 GPa (5O) and 3.13 GPa (10O).

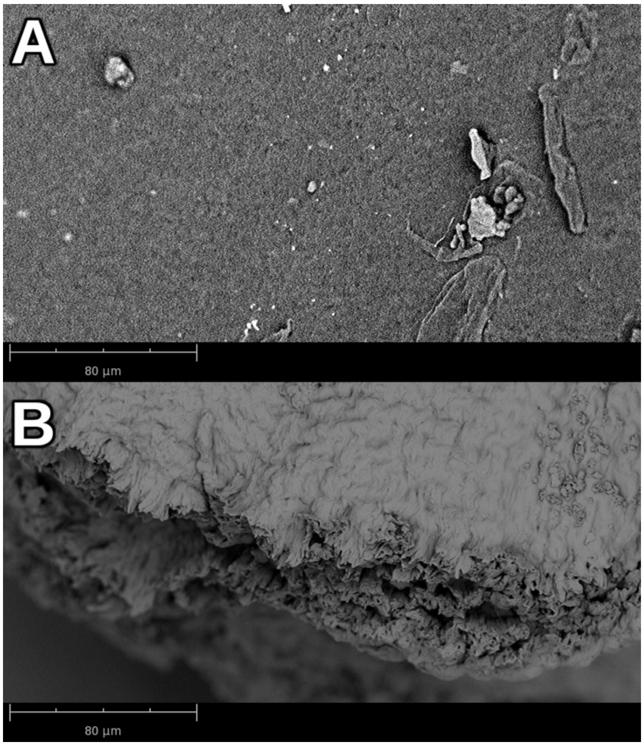

Fig. 6. SEM images of fractured sample (A) and eroded sample (B)

Figure 6 shows an SEM photo of the sample before it was stretched (A) and after it was stretched (B). In Figure 6A, the sample surface is homogeneous; no cracks, damages or discontinuities were noticed. There are filler grains of various sizes and shapes on the surface. Figure 6B shows the fracture site of the sample after the tensile test. It was noticed that there were spaces inside, which were probably remnants of air bubbles left after the gravity-casting process of the samples.

Figures 7 (coefficient friction) and 8 (wear volume) show the abrasion test results.

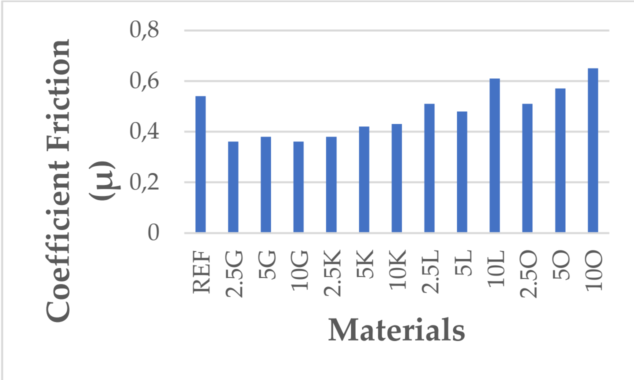

Fig. 7. Coefficient friction of tested materials

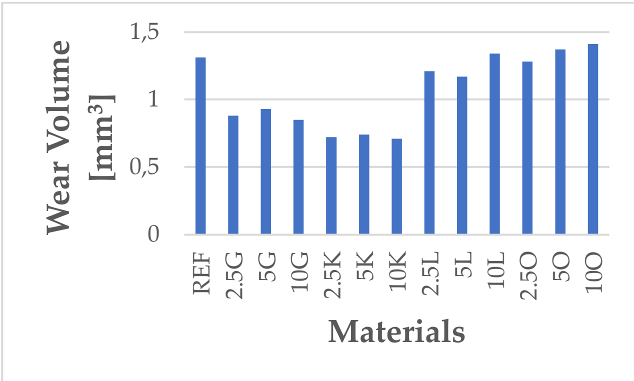

Fig. 8. Wear volume of tested materials

The coefficient friction value for neat resin was 0.54. For composite materials with grapefruit as a filler, the coefficient of friction was 0.36 for the 2.5G material, 0.38 for the 5G material and 0.36 for the 10G material, respectively. The coefficient friction values for composite materials with grapefruit filling are similar. The coefficient friction value measured for the 2.5G and 10G materials was the lowest among all tested materials and was 32% lower than the coefficient friction value for neat resin. For composite materials with key lime filling, the coefficient of friction for the 2.5K material was 0.38.

In comparison, for materials with a higher filler content, it increased and stabilised at a similar level of 0.42 (5K) and 0.43 (10K), respectively. The coefficient friction values for materials with lemon filling are 2.5L – 0.51, 5L – 0.48 and

10L – 0.61. On the other hand, for composite materials with orange as a filler, a tendency was observed that the coefficient of friction decreases with the increase in the value of the filler. The results for such a type of material are 2.5O – 0.51, 5O – 057, and 10O – 0.65. The coefficient friction value for the 10O material is the highest of all tested materials and is 20% higher than the coefficient friction value for neat resin.

The wear volume value for resin without additive for filler is 1.31 mm^3. The following results were recorded for composite materials with grapefruit filling: material 2.5G – 0.88 mm^3, 5G – 0.93 mm^3 and 10G – 0.85 mm^3. Wear volume results for composite materials with key lime filling were similar and amounted to 0.72 mm^3 (2.5K), 0.74 mm^3 (5K) and 0.71 mm^3 (10K). The wear volume value for the 10K material was the lowest among the tested materials and was 46% lower than the wear volume value measured for the REF material. In the case of composite materials with limone filler, the values were as follows: 1.21 mm^3 (2.5L), 1.17 mm^3 (5L) and 1.34 mm^3 (10L). For composite materials with orange filling, a tendency was observed that with the increase in the filler content in the composite material, the wear volume value increases. For the 2.5O material, the wear volume was 1.28 mm^3; for the 5O material – 1.37 mm^3; and for the 10O material – 1.41 mm^3. The wear volume value for the 10O material was the highest among all tested materials and was 7% higher than the value measured for the REF material.

4. Discussion

The research used ground citrus fruit peels (grapefruit, key lime, lemon and orange) as fillers for composite materials based on epoxy resin. Taking up this topic was associated with an attempt to solve two problems. The first of them concerns the problem of citrus fruit peel management. Due to their structure and the content of many waxes and toxic chemicals in the skin, they cannot be effectively recycled or composted. Citrus peels end up in landfills most of the time. In line with the postulates of the zero-waste economy, the authors found citrus peels a new use as fillers for composites based on epoxy resin. Epoxy resins have found application in many aspects of life and economy. The problem with epoxy resins is their abrasive wear associated with using products made of them and due to the forces of nature. An attempt to solve the problem of excessive abrasive wear of epoxy resins may be the addition of organic or inorganic compounds to them, which, without chemically reacting with the resin, will improve the physical properties.

Dried and ground citrus fruit peels were introduced into epoxy resin with a hardener in weight shares of 2.5, 5 and 10% by weight. Then, the resulting composite materials were gravitationally cast into moulds from which samples for testing were obtained. Without adding filler, the obtained composite materials and the resin were subjected to tensile and abrasive pin-on-disc tests.

The results of the tensile test prove that in the range of mass content of fillers from 2.5 to 10%, they do not adversely affect the mechanical properties of the obtained composite materials compared to neat resin. For the tested composite materials, it can even be seen that adding fillers does not change and, in some cases, significantly improves the mechanical properties such as peak load or peak stress. In the case of Young's modulus, however, it was observed that the effect of fillers, regardless of the concentration, is negligible and does not affect the value of this parameter. The results of the abrasive test are different. For composite materials filled with grapefruit and key lime, a decrease in both coefficient friction and wear volume was observed, regardless of the filler content in the composite. A decrease in both parameters indicates that composite materials provide the best protection against abrasive wear with grapefruit and key lime as the filler.

No significant decrease in coefficient friction and wear volume was observed for composite materials in which ground lemon and orange peels were the fillers. In contrast, for some of the tested materials, the parameters were higher than the values measured for the resin without adding fillers.

In the case of determining the abrasive wear and friction coefficient values, it was noticed that samples containing fillers such as grapefruit and key lime are characterised by lower values about the reference material (REF) and samples containing lemon and orange as fillers. The authors believe that it is because ground lemon and orange peels can form agglomerates, and thus, their consumption during grinding can be reduced. It is assumed that the abrasive wear of a composite material in which the filler grains are evenly distributed is lower than that in which the filler grains form agglomerates or, more significantly, irregular structures.

In the literature, there are few cases of testing the mechanical and tribological properties of composite materials in which organic materials of natural origin play the role of fillers. Alsadi et al. They examined the influence of the pistachio shell particle on the mechanical properties of composites whose matrix was polyester. The impact content of the pistachio shell particle in the composite was 5, 10, 15, 20, and 25 wt%. The tests showed that in the case of composites with a content of 5 and 10%, an increase in tensile strength, flexural strength, and impact strength was observed compared to polyester without adding filler. For the filler content of 15, 20 and 25%, the results of these properties were lower than for neat polyester [35]. Nayak et al. investigated the possibility of using pistachio shell particles with flax fibres as reinforcement in polyester composites. Composites with a 15% mass fraction of fibres with pistachio shell flakes range from 1% to 3%. The results indicate that adding pistachio shell flakes reduces the tensile properties of the composites while their flexural and impact properties improve [36]. Al-Obaidi et al. investigated the effect of pistachio shell particles with different grain diameters on the mechanical properties of epoxy matrix composites. The research proved that grains of pistachio shell particles of various diameters may improve, deteriorate or have no effect on tensile, flexural, Izod impact and hardness tests compared to epoxy resin [37]. Zarrinbakhsh et al. investigated the effect of coffee chaff (CC) and spent coffee grounds (SCG) on the physicochemical properties of polypropylene matrix composites. The tests showed that the introduction of 25% of the filler content into the composite significantly deteriorated the mechanical properties such as tensile yield strength, tensile modulus, elongation at yield, and elongation at break compared to polypropylene without the addition of filler [38].

5. Conclusions

The conducted research showed that the introduction of ground citrus fruit peels: grapefruit, key lime, lemon and orange to the epoxy resin caused a change in the mechanical and tribological properties of the resulting composite materials. The tensile test proved that adding organic fillers in the range of 2.5 to 10% by mass increases or does not affect the Peak Load and Peak Stress values. The tested fillers do not affect Young's modulus value compared to the resin used as a filler. The hardness test also showed no significant effect of adding ground citrus peels on the Shore D hardness value. The pin-on-disc abrasion test showed that grapefruit and key lime composite materials significantly reduced coefficient friction and wear volume. Composite materials with grapefruit and key lime as fillers are characterised by the best tribological properties with no change in mechanical properties that are not worse than the resin used as the matrix. Overall, the studies demonstrated the theoretical possibility of using fillers in the form of ground citrus peels as fillers in polymer composite materials. However, this requires further research using other materials and more advanced ageing and chemical resistance tests.

Additional information

The authors declare no conflict of interest. All authors contributed equally to this study as the co-first authors. All the co-authors have approved the final version of the manuscript.

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