

PRODUCTION OF USEFUL COMPOSITE PARTICLEBOARD FROM WASTE ORANGE PEEL

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In this study, polymeric composite particleboard has been manufactured from orange peel, which is an abundant and promising agricultural waste in Turkey. An urea-formaldehyde (95 wt%) and phenol-formaldehyde (5% wt) mixture was used as polymeric binder and waste orange peel as a filler for the production of particleboard. The effect of polymeric binder/filler ratio on the tensile strength, limit oxygen index and water absorption capacity of the composite materials were determined. The molding temperature and pressure were set to 120 °C and 2 Mton, respectively. The highest tensile strength was obtained as 15 MPa at an equal polymer/orange peel ratio. Both mechanical strength and LOI (limiting oxygen index) values increased with increasing polymeric binder amount in the particleboard. However, water absorption capacity values decreased from 0.35 g water/g material to 0.16 g water/g material, with increasing amount of the polymeric binder. Considering the properties of the developed polymeric composite material, it can be concluded that orange peel waste can be used as a filler to replace wood-based materials in the production of particleboard.

Keywords: polymeric composite particleboard, three point bending test, limiting oxygen index, recycling of agricultural wastes, orange peel

INTRODUCTION

The supply of natural resources is quite limited to meet the needs of a growing world population. The ecological balance is negatively affected as a result of carbon emissions caused by the exploitation of natural resources. For this reason, the recycling of waste materials into useful final products has become an attractive research area.

In recent years, the production of composite materials using agricultural wastes instead of wood has come into the research focus. High mechanical and physical properties, biodegradability, waterproof and fireproof properties, as well as low cost, are required features, so that polymeric composite materials can compete with wood-based materials.

Polymeric composite particleboard is formed by combining a polymeric binder and filling materials under appropriate conditions and ratios. Most commonly used polymeric binders in the production of particleboard are urea formaldehyde,¹⁻³ phenol formaldehyde,⁴ polyethylene^{5,6} and polyvinyl acetate.^{7,8} It is expected that these polymeric binders may increase the mechanical strength, waterproofing, fireproofing and biodegradation resistance of composite materials.

The assessment of agricultural wastes, which have low calorific value, to be used as a filling material in manufacturing commercial particleboard, is important to reduce the dependence on trees as a wood source. Boards prepared from various agricultural wastes have different physical properties depending on their specific cellulosic structure. It is important to evaluate agricultural wastes to find their particular characteristics, which may also vary as a function of the geographical area where the plant has grown. A wide variety of non-wood plant fibers and agro-residues, such as nutshell,¹ walnut and almond shell,⁹⁻¹¹ peach nut shell,¹² kiwi prunings,¹³ bamboo,¹⁴ cotton seed hulls,¹⁵ flax shives,¹⁶ vine prunings,¹⁷ wood flour,¹⁸ rice husk and starch,^{19,20} Todo fir, sycamore leaves and sun flower, have been investigated as filling materials.²¹⁻²³

In the literature, studies on a number of parameters, such as curing time and temperature of the polymeric binder,^{1,9} molding temperature and pressure, polymeric binder/filler ratio^{10,12} and particle size of the filler,²⁴ have been carried out to find the optimum conditions for resin synthesis and to enhance the physical and mechanical properties of particleboard.

Guru *et al.* have used almond shell, which has lower calorific value, as a filling material in the development of polymeric composite particleboard in their study. The effect of different urea/formaldehyde ratio, polymerization temperature and particle size of filler has been investigated. The tensile strength of the material that was obtained at the urea/formaldehyde ratio of 0.97, 70 °C polymerization temperature, 25 minutes reaction time and 0.3 mm mean particle size was determined as 84.5 N/cm².¹⁰

In our previous study, we used peach nut shell and phenol formaldehyde to produce particleboard. The optimum conditions to obtain the highest tensile strength have been determined. As a result of experimental work, the best tensile strength of the particleboards were obtained as 34MPa at 2.72 MPa molding pressure and 120 °C molding temperature, with 150 µm particle size of the filling material.¹²

In some studies, more than one polymeric binder and filling material were used in different ratios and their effects on the mechanical properties of particleboards were examined. High-performance lignocellulosic hybrid composite board was developed by using a mixture of rice straw and coconut fiber. The effect of the rice straw/coconut fiber ratio on the tensile strength and the increment in thickness was investigated. A NaOH solution was used to remove the silica layer from the rice straw. It was determined that a higher rate of coconut fiber has a negative effect on the mechanical strength and the increment in thickness.⁵

Different walnut–almond shell particle ratios (0-100%) were investigated using urea–formaldehyde as binder. The moduli of elasticity and rupture, internal bond strength, thickness swelling and water absorption of the particleboard were evaluated. It was reported that the addition of walnut–almond particles significantly improved the water resistance of the panels. On the other hand, increasing the walnut–almond shell content in the panels reduced the flexural properties and the internal bond strength.¹¹

In the study of Ayırmis *et al.*, the effects of resin type were investigated on the dimensional stability and mechanical properties of single-layer composite particleboards made from a mixture of wood particles (70 wt%) and rice husk particles (30 wt%). Urea–formaldehyde (UF) and phenol–formaldehyde (PF) resins, in different ratios, were used. It was reported that the mechanical properties of the PF resin bonded samples were better than those of the UF resin-bonded samples.²⁵

In the study of Klimek, the effects of both filler materials and resins were investigated. Germany-grown cup-plant (*Silphium perfoliatum* L.), sunflower (*Helianthus annuus* L.) and topinambour (*Helianthus tuberosus* L.) were used as raw materials, while methylene diphenyl diisocyanate (MDI) and urea formaldehyde (UF) were used as resins to produce particleboard. They concluded that the MDI-bonded particleboard made from agricultural residues was a viable alternative to classical UF-bonded particleboards.²³

Fireproofing is required for wood-based materials. This feature has been investigated using various additives. Cavdar examined the effect of wood preservatives, with different chemical loadings, on the fire performance of wood, by the oxygen index test technique. The researcher concluded that all the treated samples had higher LOI (limiting oxygen index) values than the untreated ones.²⁶ In another study, the potential of poppy husk for manufacturing wood based particleboards was examined. The researchers found the LOI values for 100% poppy husks and 100% pine woods were 48 and 36, respectively.²⁷ In our previous studies, fly ash and glass powder were used to enhance the fireproof ability of particleboards produced from pistachio and peach nut shells. The addition of 20 wt% fly ash reduced the flame temperature of the particleboards manufactured from pistachio shells from 795 K to 568 K. Also, glass powder, varying between 0-50 wt%, was added to particleboards produced from peach nut shell. The LOI values were enhanced from 41 to 50 upon the addition of 30% glass powder.^{4,12}

Particleboards emit volatile organic compounds (VOCs), which can be harmful to human health. Formaldehyde-based resins, such as urea formaldehyde and phenol formaldehyde, are often used for producing particleboards and their VOCs are known as carcinogenic.²⁸⁻³¹ Therefore, formaldehyde emissions from wood materials have become a topic of great research interest. For example, Kim *et al.* tried to determine the formaldehyde and VOC emissions from wood-based composites, such as medium density fiberboard (MDF) and particleboard (PB), by the desiccator and perforator methods. They reported that the formaldehyde emission of MDF and PB was 3.48 mg/L and 5.38 mg/L by the desiccator method, and 8.57/100 g and 10.21/100 g by the perforator method, respectively.³²

The emission characteristics of formaldehyde and other VOCs from particleboard in sealed or ventilated environmental chambers were examined at different temperatures (23, 35 or 50 °C). It was reported that the emissions of formaldehyde and total VOCs from the particleboard increased significantly with rising temperature.²⁹

Liang *et al.* examined the effect of humidity on formaldehyde emissions from MDF and suggested a possible mechanism. They reported that the effect of humidity on formaldehyde emissions was based on the hydrolysis of resins or polymers and on the adsorption competition between formaldehyde and water molecules.³³

In the study of Ghafari *et al.*, furfural was used instead of formaldehyde and the changes in the physical-mechanical properties and formaldehyde emission of particleboard were investigated. They established that formaldehyde emissions and the modulus of rupture (MOR) of the panels reduced upon replacing the formaldehyde with furfural in the UF resin. A temperature increase from 170 °C to 180 °C increased the formaldehyde emissions.³⁴

Additive manufacturing, also named as 3D printing, is emerging as a new field of work in the production of wood materials. Because of the disadvantages of 3D printing, such as using expensive materials and negative environmental impact, 3D printing processes, such as fused deposition modeling (FDM), selective laser sintering (SLS), stereo lithography (SL), laminated object manufacturing (LOM) and shape deposition manufacturing (SDM), have been investigated to overcome these disadvantages. All these processes are related to rapid prototyping (RP), which is the technology used for reducing product development time and production cost.³⁵⁻³⁹ Garg *et al.* studied genetic programming (GP), support vector regression (SVR) and artificial neural networks (ANN), which are soft computing methods for improving the environmental performance of the SLS process. They reported that GP was the best method for predicting open porosity based on laser power data.³⁸ In another work, the mechanical properties of polyamide were evaluated by using SLS. Energy density was investigated as a function of laser beam speed and average power. It was reported that the laser power had a higher effect than scan speed on density.³⁹

Complex pieces, which are virtually impossible to manufacture by conventional processes, can be produced easily using these methods. Some polymers, such as polyamide, acrylonitrile butadiene styrene (ABS), polylactic acid (PLA) and polyvinyl alcohol (PVA), can be used for 3D printing.

PLA is mostly used in 3D printing process and its specific tensile and flexural mechanical properties have been investigated. Hinchcliffe *et al.* examined the effect of additive manufacturing (3D printing) and initial post-tensioning of continuous natural-fiber reinforcement on the mechanical properties of PLA. They reported that 3D printing can improve the specific tensile and flexural mechanical properties of PLA composites.⁴⁰

Wood powder and mixtures of adhesives, including polyvinyl acetate and urea formaldehyde (UF), have been used for 3D printing. Experimental results showed that wood powder could be used for additive manufacturing. 3D printed parts with UF had better properties than those with polyvinyl acetate.³⁶

A literature survey reveals that many agricultural wastes have been used as alternatives for commercial wood-based products. However, while orange peel is a waste resulting in considerable quantities from fruit juice factories in Turkey, there is no work on the use of orange peel as a filling material. The aim of this study was to investigate the development of particleboards with high mechanical strength, fireproof and waterproof ability from orange peel. A urea-phenol formaldehyde resin mixture was synthesized and used as a binder. The particleboards were prepared with different resin and orange peel ratios (1/1, 1/2, 1/3, 1/4). The performance characteristics of the composite particleboards were determined by the three point bending test, water absorption capacity test, impact test, hardness test and fireproofing test (LOI).

EXPERIMENTAL

Materials and methods

The preparation of polymeric composite particleboard from orange peel was carried out in two steps, as described in Figure 1. The first step consists in the synthesis of the polymeric binder and the second – in the production of the polymeric composite material by mixing the filling material and the binder. A formaldehyde solution (37% (w/w), Sigma Aldrich), H₂SO₄ (97% (w/w), Sigma Aldrich), phenol (99.9% (w/w), Carlo-Erba)

and urea (99% (w/w), Merck) were used in the synthesis of the polymeric binder. H_2SO_4 was used as a catalyst in the synthesis of the polymeric binder.

The urea formaldehyde and phenol formaldehyde resins were prepared in a flask reactor, with reflux condenser, thermometer and magnetic stirrer. A 500 mL volumetric flask was used as polymerization reactor. For the preparation of the urea resin, urea, formaldehyde and NaOH catalyst were added into the flask and heated up to 50 °C; the reaction temperature was controlled within ± 2 °C. The mixture was mixed until it became homogeneous. After the polymerization reaction was completed at the specified temperature and time, the filler material was added into the polymer.

The polymeric resins were prepared as described in earlier studies.^{1,4,9} The phenol formaldehyde resin was prepared by the condensation of phenol in formaldehyde at 70 °C. A H_2SO_4 solution was added into the phenol formaldehyde solution for curing. The phenol/formaldehyde ratio was set to 1 and thorough mixing was provided. The temperature was fixed at 90 °C and the polymerization was carried out. 95% by mass urea resin and 5% by mass phenolic resin were mixed and homogenized.

Orange peel, the filling material, was dried at 70 °C until constant mass was reached. It was then ground to 150 μm particle size, using a Retsch SM100. Different resin/filling material ratios (1/1, 1/2, 1/3, 1/4) were used for preparing particleboard. The filling material–polymeric resin mixture was molded and pressed at 120 °C and 2 metric tonnes using a Carver hot press unit.¹⁹ In order to remove the residual volatiles, the particleboard was then kept at 70 °C for 4 hours. The dimensions of the prepared particleboards were 120 mm length, 60 mm width and 10 mm thickness.

The mechanical strength of the particleboards was determined by the three point bending test according to EN 310 standard, using a Shimadzu AG-I apparatus. The dimensions of the rectangular specimens, which was cut from the prepared samples, were 10 mm thickness, 12 mm width and 60 mm length, as illustrated in Figure 2. The hardness and impact tests of the particleboard were performed using Emcotest Duravision and Brooks devices. The hardness test was carried out according to ASTM E92-17 standard. In this test, samples of the same size as for the bending test were used. Vickers hardness tests were carried out in the temperature range from 10 to 35 °C. The indenter was cleaned; the test force corresponded to HV10 Vickers scale and was applied for approximately 10 s. The impact test was carried out according to ASTM E23 standard. The specimen was cut to 55 mm length, 10 mm width and 10 mm thickness. Zero position of the machine was set and the notch was made in the middle of the specimen.

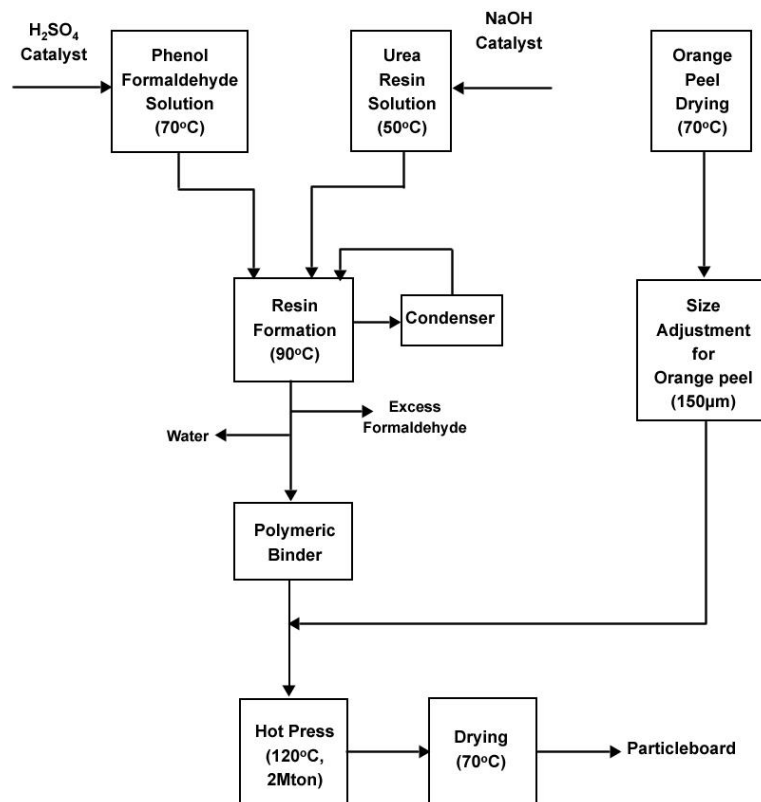


Figure 1: Preparation of polymeric composite particleboard from orange peel

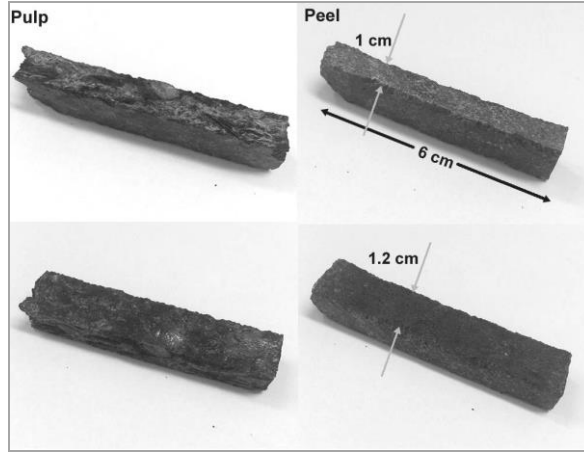


Figure 2: Rectangular specimens of polymeric composite particleboard from orange peel and pulp

The limiting oxygen index (LOI) of the samples was measured using a LOI device (DYNISCO Polymer Test) according to ASTM D2863 standards. In this analysis, a sample of 100 mm length, 10 mm width and 5 mm thickness was used. The test sample was positioned vertically in a glass chimney and an oxygen/nitrogen environment was established with a flow from the bottom of the chimney. The top of the sample was ignited and oxygen concentration in the flow was increased until a continuous flame was obtained for 30 seconds. To determine the water absorption capacity and swelling specifications of the particleboards, the materials were held in distilled water for 24 hours. The water absorption capacities of the particleboards were determined by using Equation 1:

$$\text{Water Absorption Capacity} = (m_{\text{wet}} - m_{\text{dry}}) / (m_{\text{dry}}), \text{ g water/g materials} \quad (1)$$

where m_{wet} – weight of wet material, g; m_{dry} – weight of dry material, g.

Also, the thickness change was determined using Equation 2:

$$\% \text{ Thickness} = (t_{\text{wet}} - t_{\text{dry}}) / (t_{\text{dry}}) * 100 \quad (2)$$

where t_{wet} – thickness of wet material, cm; t_{dry} – thickness of dry material, cm.

The experiments for the water absorption capacity and swelling properties of the particleboards were carried out according to EN 317 and ASTM-D 1037 standards. SEM analysis was also performed to understand the morphological structure of the particleboard using a JEOL JSM-6360.

RESULTS AND DISCUSSION

In this study, particleboards were produced with different ratios of filling material/polymeric binder, using orange peel as a filling material. The mechanical strength, water absorption capacity and LOI value of the particleboard were determined.

The pulp part accounts for about 10-20% of the mass of waste orange peel. Therefore, it has been investigated whether it can be used directly in the production of composite materials, taking into account its availability in considerable quantities. Particleboards from orange pulp and peel were produced using a binder/filling material ratio of 1/2 at 2 MPa molding pressure and 120 °C molding temperature. Their mechanical strengths were compared in Table 1. It was seen that the particleboard produced from orange peel has higher impact resistance (13 MPa) than the particleboard produced from orange pulp (4 MPa). There may be several reasons why the particleboards prepared using orange pulp have lower mechanical strength than those prepared using orange peel. It is possible that the moisture from the pulp could not be completely removed. Also, the pulp was used directly without grinding and this could cause poor dispersion in the structure. Therefore, further experimental studies were performed using only the orange peel particleboard due to its higher tensile strength.

Figure 3 shows the tensile strength values obtained from the three-point bending test for materials with different polymeric binder/filler ratios.

Table 1
Tensile strength of composite particleboard prepared based on orange pulp and orange peel

Filler material	Polymeric binder/filler ratio	Tensile strength (MPa)
Peel	1/2	13
Pulp	1/2	4

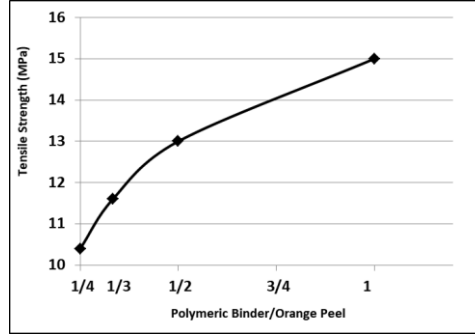


Figure 3: Changes in tensile strength as a function of polymeric binder/orange peel ratio

Table 2
Vickers hardness and impact values of polymeric composite particleboard

Polymeric binder/filler ratio	Vickers hardness	Impact (J)
1/4	23.6	1.1
1/3	24.1	1.2
1/2	25.2	1.2
1/1	25.9	1.3

As seen from Figure 3, tensile strength increased when the amount of polymeric binder was increased. It was determined that the particleboard reached saturation at an equal polymeric binder/filling material ratio. When the amount of the polymer was increased (polymeric binder/filling material ratio >1), a higher amount of polymer leaked from the mold.

Gürü *et al.* produced particleboard based on walnut shell with different ratios of walnut shell/polymeric resin. They identified that when the proportion of the resin in the particleboard was increased, the tensile strength also enhanced. The tensile strengths of 3.8 Mpa and 1 MPa were obtained for the ratios of walnut shell/polymeric resin of 3/1 and 7/1, respectively.⁹ Sahin *et al.* also investigated the effect of polymeric resin/filler ratio on the tensile strength. They reported that the varying the filler/polymeric resin ratio from 1/1 to 7/1 decreased the tensile strength from 18.5 MPa to 7.3 MPa.¹²

Vickers hardness and impact test values of the prepared polymeric composite particleboards are given in Table 2. The highest Vickers hardness value (25.9) was recorded for the particleboard formulation with the highest tensile strength, indicating that the particleboard contains a soft material. Besides, the impact test for the same composite resulted in a value of 1.3 J. It was observed that the impact value of the particleboard was quite low, compared with those of titanium (24.9 J) and aluminium (6 J) alloys.^{41,42}

In Figure 4, the variation in the water absorption capacities of the composite materials prepared with different polymer/orange peel ratios over time is shown. After 15 hours, there is no significant change in the water absorption capacities of the polymeric materials.

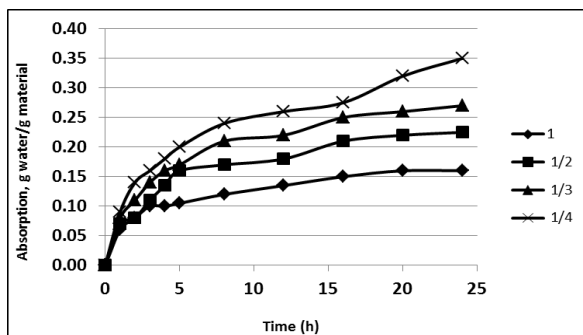


Figure 4: Water absorption capacity *versus* time for the developed composite particleboards

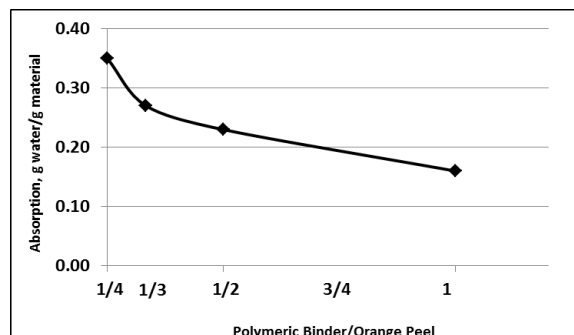


Figure 5: Water absorption capacities of composite materials with different polymer/orange peel ratios

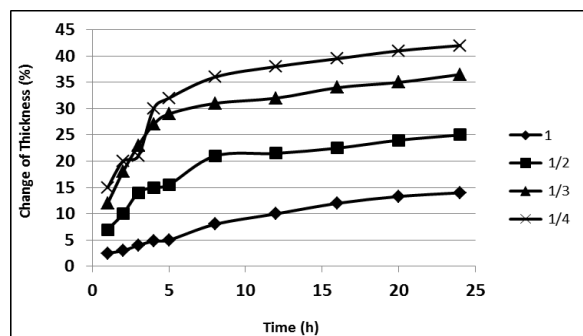


Figure 6: Change of thickness *versus* time for the developed composite particleboards

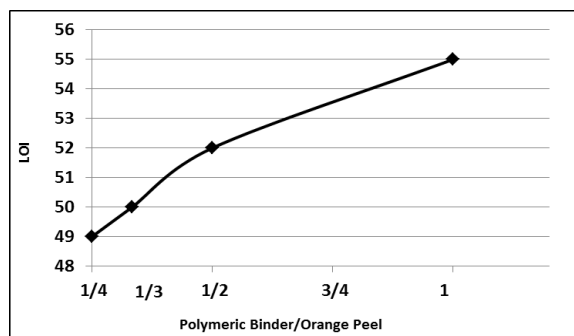


Figure 7: LOI values of composite materials with different polymer/orange peel ratios

However, the material with the lowest polymer ratio (1/4) did not show the same constant profile like the other materials. This can be explained by the fact that because of the the high ratio of orange peel and the low amount of polymer, the filler material was not sufficiently coated with the polymer. Considering that orange peel is hydrophilic, such an increase in the water absorption capacity could be expected.

In Figure 5, the water absorption capacities of the composite materials with different polymer/orange peel ratios is shown. It is observed that the water absorption capacity decreases with increasing polymer amount. When the polymer/orange peel ratio is 1/4, the absorption value is approximately 0.35 g water/g material and this value decreases by half for the particleboard with the polymer/orange peel ratio of 1/1. The polymeric resin consists of 95% urea resin and 5% phenol resin. Other studies in which phenol and urea resin were used together have shown that with increasing the proportion of phenolic resin, the water absorption and change in thickness values decreased.¹⁹ In our previous study, the water absorption capacity of particleboard prepared using only phenol formaldehyde resin, 150 μ m average particle size and 1/3 polymer/filler ratio was found to be of 0.016 g water/g material.¹² In this study, the absorption capacity obtained was nearly 0.27 g water/g material. This considerable difference in the water absorption capacity is thought to be caused by the resin used more than by the filler. Similar water absorption capacity results were obtained in some other studies in which urea formaldehyde resin was used.⁴³⁻⁴⁵ In the literature, it has been reported that phenol formaldehyde resin is more durable than urea formaldehyde resin. This type of resin provides very good resistance to moisture, which prevents excessive moisture absorption.⁴⁶ Considering that the phenolic resin in the particleboard is hydrophobic, it is expected that with an increasing polymer ratio in the material, the water absorbed would decrease.

The change in thickness of the composite materials developed using orange peel over time and for different polymer/orange peel ratios is illustrated in Figure 6. As a result, as the polymer ratio increases in the material, the change in thickness decreases, similarly to the water absorption capacity trend.

In Figure 7, the LOI values of the developed polymeric materials are provided. With increasing polymeric binder/orange peel ratio, the LOI value also increases significantly. As specified in ASTM standards, materials with LOI values over 21 are accepted as non-flammable. Thus, according to the results, all of the materials developed in the present study are non-flammable. The LOI value of pure

dried orange peel was determined as 21. As the orange peel ratio increases in the composition, due to the inflammability of totally dried orange peels, the LOI value decreases. Baishya *et al.* investigated the effects of different cross-linkers on the properties of starch/wood composites. They reported that the flame retardancy of the composites was improved due to cross-linking between the urea formaldehyde and phenol formaldehyde resins.⁴⁷ Urea formaldehyde and phenol formaldehyde resins are known as cross-linked thermoset materials.^{47,48} Thus, the flame retardancy of the composites was improved due to the polymeric resin adhesives. A higher amount of polymer resin in the formulation contributed to better flame retardancy of the particleboards due to properties of the polymeric binders.

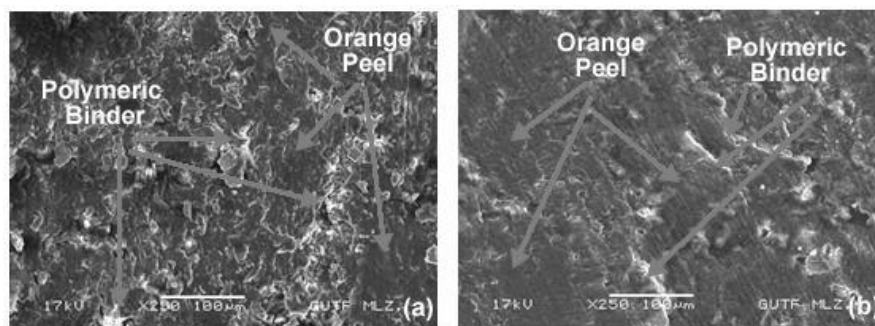


Figure 8: SEM images of composite materials; (a) vertical section, and (b) horizontal section

SEM images of horizontal and vertical sections of the composite material prepared with the polymeric binder/orange peel ratio of 1/1 are given in Figure 8. As seen from the figure, the polymeric binder and the orange peel parts of the composite particleboard are distributed homogenously.

CONCLUSION

Our literature survey has revealed that the usage of orange peel as filler in the manufacture of particleboard has not been investigated so far. As orange peel is produced as a waste by juice plants in substantial amounts in Turkey, in this study, composite particleboards were developed based on orange peel and the potential of orange peel to replace wood in particleboards was analyzed. The mechanical and chemical properties, as well as the flammability performance of the developed particleboard were evaluated. Urea formaldehyde and phenol formaldehyde mixtures were used as binding agent.

The tensile strengths, water absorption capacities, change in thickness and LOI values of the particleboards were determined. The results showed that the tensile strength of the particleboards increased by increasing the polymer ratio in the formulation and the highest tensile strength of 15 MPa was obtained for an equal polymer/filler ratio. The increase in the amount of polymeric binder in the composition also enhanced the non-flammability (LOI) of the particleboards. However, the water absorption capacity significantly decreased from 0.35 to 0.16 g water/g material as the polymer/filler ratio was varied from 1/4 to 1/1. In addition, it has been found that the polymeric resin used in the production of particleboards has a considerable effect on the water absorption capacity of the material.

Commercially available conventional wood-based materials have some disadvantages, such as flammability, degradation and swelling. In this work, the flammability and swelling properties of the particleboard produced from orange peel were enhanced by an increase in the amount of polymeric resin used. Thus, it could be possible to prevent the degradation of particleboard by controlling the binder content.

To conclude, the developed polymeric composite particleboards by using urea-formaldehyde and phenol-formaldehyde and orange peels can be considered as a substitute for commercial wood-based materials (chipboard, *etc.*), owing to their higher tensile strengths, higher water resistance and non-flammability.

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