

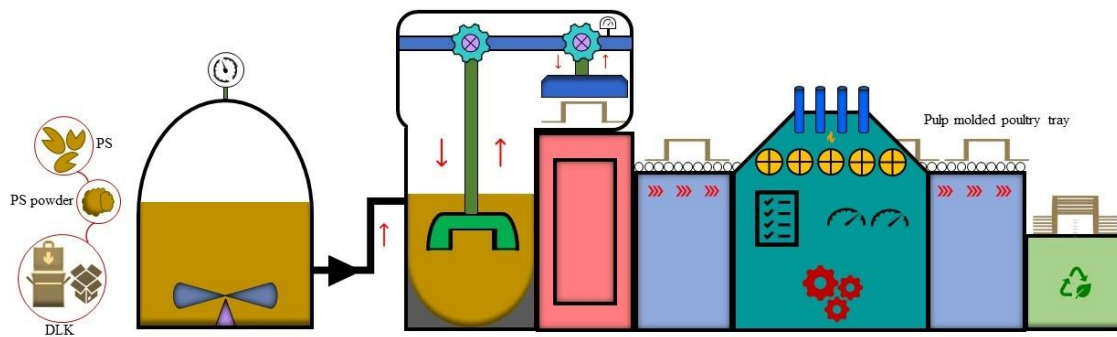
Pistachio Shell Powder as an Additive in Molded Pulp Products

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GRAPHICAL ABSTRACT



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Molded pulp products (MPP) products can accommodate a variety of fiber types and additives, including agricultural residues. This work showed the industrial scale production of molded pulp food trays made of double lined kraft clipping fibers with the addition of pistachio shell (PS) powder at levels from 5 to 30 wt% and evaluated the mechanical and barrier properties of the trays. The PS powder used to make the trays was ground using an impact mill and a cryo mill to a D50 of approximately 50 microns. Tray compression strength was tested to simulate the wrapping of food trays with plastic wrap. Additionally, the tray material's basis weight, thickness, density, tensile strength, short span compressive strength, and absorptiveness were determined. The results showed that compared to the control tray sample, the trays containing the cryo-milled pistachio powder had essentially the same engineering properties as the control trays without PS. For the trays made with the impact-milled powder, the tensile strength, bending resistance, and compressive strength increased relative to the control 11.3%, 6.2%, and 13.5%, respectively. The study demonstrates a method to evaluate the incorporation of alternative materials into pulp molded products.

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Keywords: Pulp molded food tray; Pistachio shell; Agricultural waste; Recycled fiber (double-lined kraft)

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INTRODUCTION

Generally, there is now greater emphasis on employing environment-centric solutions and preventing the destruction and overexploitation of natural resources than ever before (Lee *et al.* 2024; Mirzaei *et al.* 2024). In recent decades, eco-based and cost-effective manufacturing concepts have led to the next generation of bio-based composite materials (Johansson *et al.* 2012; Li *et al.* 2023). Food packaging traditionally consists of synthetic plastic packaging made from fossil sources, which can take many years to degrade and puts a great deal of pressure on the environment (Fereidooni *et al.* 2024). A variety of policies and incentives have been implemented by many countries to encourage the use of plant-based fiber packaging that is recyclable, reusable, and compostable (Zhang *et al.* 2022; Vinod *et al.* 2023). The food packaging industry accounts for over 40% of all industrial packaging (a global market valued at over \$56 billion), with plastic packaging representing 40% of all food packages (Rattanawongkun *et al.* 2020).

As a method to recycle paper, cardboard, and wood pulp, molded pulp products are a relatively mature industry that was developed in the late 19th century (Semple *et al.* 2022). Many companies and packaging industries are now using molded pulp products as an environmentally friendly alternative to plastics and less sustainable materials (Gouw *et al.* 2017; Elhussieny *et al.* 2020). As a potential input to pulp molded products, about 140 billion tons of forest and agricultural waste including wood and plants are produced worldwide each year that are cheap, renewable, and abundant (Cheng 2017; Liu *et al.* 2017; Cortat *et al.* 2021a). These residues can be valorized by transformation into biosorbent materials that are practical and economical due to the biomass surface's functional groups and porosity (Leichtweis *et al.* 2020). Agricultural waste can be used in the fabrication of green composites as a natural filler, due to their cellulosic structure (Kuram 2021). Palm and coir, nutshells, kenaf fiber, olive stone, rice husk, bagasse, peanut shell, jute, and sugarcane have been used in polymer matrices composites as fillers for reducing the production costs as well as enhancing the mechanical and physical properties (Maghsoudi *et al.* 2010; Sareena *et al.* 2012).

Nutshells are one of the materials utilized as a filler in plastic matrix composites, because of their cellulosic composition that can contribute to the composite properties (Prabhakar *et al.* 2015; Boran Torun *et al.* 2021). Nutshells are agricultural byproducts of nut processing and are renewable lignocellulosic materials, which have roughly 30 to 40% lignin and 25 to 30% hemicellulose and cellulose (Copur *et al.* 2007; Sutivisedsak *et al.* 2012; Qiang *et al.* 2022). Macadamia is a widely-utilized nut, which has high amounts of shell residues. Cortat *et al.* (2021b) presented a method for green composites development by macadamia nutshell residues (MR) addition. Various MR contents, ranging from 5 to 30 wt%, were combined with polypropylene (PP) composites as a filler. The thermal analysis outcomes demonstrated a significant improvement in the thermal stability of PP by adding MR. This enhancement is probably because of the high amount of lignin present in the filler. Life cycle assessment (LCA) showed lower environmental effects, such as terrestrial acidification, climate change, fossil depletion, and photochemical oxidation, by increasing MR contents in comparison with the classical handling of this residue.

Silva *et al.* (2021) studied the mechanical and thermal properties of polypropylene made with 5 to 30 wt% macadamia nutshell fiber with an alkaline treatment. The composites had higher thermal stability at higher temperatures. Additionally, by adding treated and raw macadamia fibers to PP, the stiffness was enhanced. The integration of 30 wt% treated macadamia nut fiber to PP produced a 67.5% increase in the tensile modulus. Song *et al.* (2020) prepared poly lactic acid (PLA) with almond, walnut, wild almond, and macadamia shell powder (ASP, WSP, WASP, and MSP, respectively). For PLA/nutshell composite filaments preparation, the fused deposition modeling technology was utilized. The highest degradation temperature was realized using the wild almond shell powder treatment of PLA. In comparison with the simple PLA, the PLA treated with macadamia and WASP improved the degree of crystallization. Additionally, the investigation related to tensile strengths showed that the PLA treated by WASP-Na and MSP-Na (Na indicates the NaOH treatment) had no significant effect relative to PLA. Furthermore, studies on the water-resistance properties presented that the MSP treatment on the PLA had the best performance relative to the other powders.

Pistachio shells represent a large annual volume of agro-industrial remnants in Turkey, the United States, Iran, and many other countries (Balasundar *et al.* 2019).

According to the Food and Agriculture Organization (FAO), the yearly production of pistachio shells in the United States was about 523,900 tons in 2021 (FAO 2023). The addition of this material in polymer composite constructions is appropriate for engineering applications that require adequate tensile strength, elastic modulus, and the other properties (Akovali 2001). In this regard, Balasundar *et al.* (2019) reported that pistachio nutshells possess good thermal and structural properties for bio-filling usage. An abundant agro-industrial waste product with little commercial usage, pistachio shells offer a sustainable and reasonably priced alternative for developing new uses.

Given more conventional, softer lignocellulosic fillers, the special mechanical qualities of PSP-including its hardness and density-were considered to possibly improve the mechanical properties of the molded pulp products, especially in terms of compression strength. Moreover, Gürü *et al.* (2009) produced a construction material using urea-formaldehyde and pistachio shells and enhanced its non-flammable characteristics by adding fly ash.

For composite particleboard construction, crushed pistachio shells and urea-formaldehyde were mixed at various ratios from 0.66 to 1.22 (w/w) urea-formaldehyde/pistachio shells and dried for 24 h at 60 °C in an oven. In another investigation, Alsaadi *et al.* (2018) studied the effect of microscale particles of pistachio shells on the mechanical properties of polyester matrix composites. Flexural modulus, Charpy impact, and tensile tests were carried out on the molded composite samples. Various contents of pistachio shell particles (PSP) were applied, and the results demonstrated that the maximum impact strength, tensile strength, and flexural strength were obtained at 5, 10, and 25 wt% of PSP content, respectively. Considering the interest and amount of research involved in the usage of different nutshells in composites, it was deemed of interest to evaluate the application of PSP into pulp molded (fiber-based) products.

The objective of this paper was to provide information regarding the production of pulp molded food trays (suitable, for example, to hold poultry, as a poultry tray) containing different percentages of PSP. To the best of the authors' knowledge, this is the first work reporting the use of PSP in the production of pulp molded trays. Using an industrial-sized pulp molding machine to form, press, and dry the parts, food trays containing different percentages of pistachio shell powder were produced at real production rates. The chemical composition and mechanical characteristics of each pulp molded food tray were examined. The trays were characterized using X-ray Diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), particle size distribution, Scanning Electron Microscopy (SEM), Energy Dispersive X-ray spectroscopy (EDS), and CHNO analysis. Additionally, the pulp molded food trays were tested for their basis weight, thickness, density, tensile strength, stretch, tensile index, Cobb test, and short span test. It is shown that PSP can be incorporated into these types of food trays with acceptable properties, producing valuable products from this agricultural residue.

EXPERIMENTAL

Materials

The Wonderful Company provided pistachio shells for this research, originating from California, USA.

Table 1. Chemicals in MPP Enhanced with PSP as an Additive

| Product Name | Product Description | Key Benefits |
|--------------------|---|--|
| TopScreen MF305-NA | Hot oil holdout | FDA compliant for food packaging, Non-PFAS |
| Hercon 615 | Sizing agent (AKD) | FDA compliant for food packaging, works with both bleached and unbleached pulps |
| Hercobond 6950 | Highly cationic charged Polyvinylamine (PVAM) | FDA compliant for food packaging, improves dry strength, drainage, and retention |
| Perform SP7242 | Anionic water-soluble polymer | FDA compliant for food packaging, drainage, retention, and clarification |

Preparation and Specification of the Pulp Molded Trays

Industrial scale trials to produce pulp molded food trays (suitable for holding poultry) made from recycled fiber (pre-consumer double-lined kraft (DLK)) and ground pistachio shells were conducted. The food trays were manufactured with a type 3, hot press thermoformer. A simplified schematic is shown in Fig. 1.

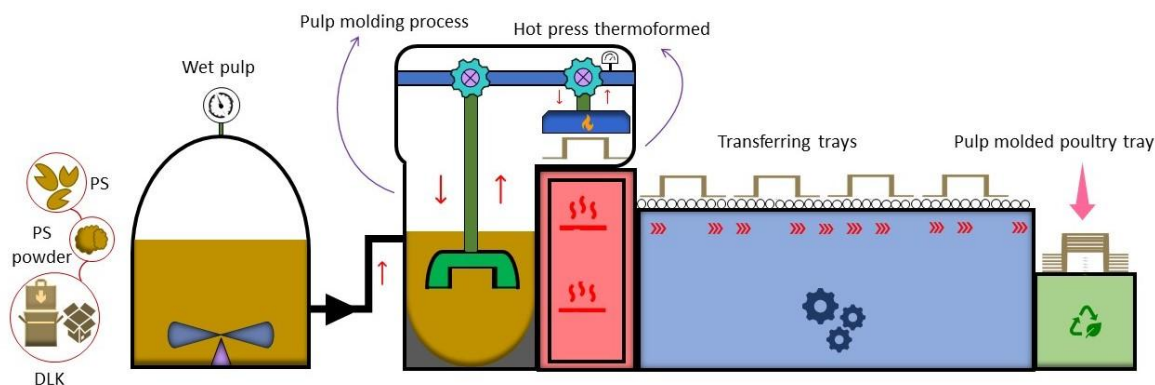


Fig. 1. Industrial scale schematic outline for molding pulp trays from recycled fiber waste DLK and PS powder

In compression molding, different types of food trays with different percentages of pistachio powder were prepared under a series of processes including pulp preparation, forming, pressing, and drying. Preparation of the DLK included collection, sorting, pulping, deflaking, and refining. The applied pulp molding machine was Taiwan Pulp Molding Co. model TPM-850 (type III). During the pulping process, the fiber raw materials were mixed with hot water in a pulper and pulped at low/medium consistency for a set time period. As a result of hydrodynamic disintegration, these fibers were separated and

detached from one another. The DLK pulp was then refined with a conical refiner and then delivered to the pulp molding machine at 0.50 % consistency. The feed tank to the pulp molded vat was equipped with an extra recirculation line as an added method to keep additives/materials well mixed in the tank. A chemical additive package, free of per- and polyfluoroalkyl substances (PFAS), was used to provide water/grease resistance and to retain small particles in the product while aiding water drainage when forming the product. The addition order of the chemicals and the PSP and their details are as follows:

1. TopScreen MF305-NA: A wax emulsion designed to improve oil holdout properties in molded fiber articles by creating a physical barrier. It is designed to work at hotter press temperatures because it is a high melting point wax. It is a Non-PFAS product with FDA compliance for food contact and formulated using compostable raw materials.

It is true that numerous traditional waxes, such as paraffin, can dissolve in oil-based liquids, which hinders their ability to provide oil resistance. However, the wax emulsion used in the present study was specifically created to enhance the oil retention in molded pulp items. The important key to the effectiveness of this wax emulsion lies in its high melting point and its ability to create a physical barrier on the surface of the fibers within the pulp matrix. This emulsion is designed to provide a strong, hydrophobic coating that opposes oil and other liquid penetration unlike other waxes. The distinct structure of the emulsion and the way it adheres when desiccated, increase its durability against dissolving in oil-based substances (Lang et al. 2022). Furthermore, the emulsion is specifically crafted to endure the high temperatures typically used in thermoforming processes, helping to create a durable protective barrier. A strong and stable network resulting from the interaction of the emulsion with the cellulose fibers and the other formulation components would efficiently stop oil penetration. Therefore, while traditional waxes might be soluble in oils, the particular formulation and using this wax emulsion provide the necessary oil holdout properties required for food tray application.

2. Hercon 615: A sizing agent emulsion based on alkyl ketene dimer (AKD) chemistry, the product reacts directly with cellulose to provide sizing with a hydrophilic head that bonds with the cellulose and a hydrophobic tail that repels water. It also contributes to drainage and retention by balancing cationic charge to bond efficiently to the anionic fibers.
3. Pistachio Shell Powder (PSP): PSP increases the molded pulp article density and performance by acting as a filler in the pulp fiber matrix, reduce fiber consumption, and commercially utilize an agricultural waste product with no current commercial benefit.
4. Hercobond 6950: A low molecular weight highly cationic charged poly(vinyl-amine) (PVAm) that improves dry strength, retention, formation, and sizing through optimizing the charge of the system to improve efficiency of the other chemistry in the system. The Hercobond 6950 works by attaching to high surface area, high anionic charged fines in the system.
5. PerForm SP7242: A retention, drainage, and clarification aid that is an organic polymer that provides superior retention, drainage, and formation for neutral/ alkaline paper and

paperboard. PerForm SP7242 is an anionic water soluble, polymer matrix in emulsion form. The high charge density and three-dimensional matrix structure enables the product to be very efficient at capturing fines, filler, developing a consistent micro-floc and enabling high levels of drainage without negatively impacting fiber formation in the forming section of the molded pulp thermoformer.

The emulsion was meticulously diluted and mixed under specified conditions to create a homogenous mixture before adding to the pulp mix. This stage was needed to confirm that the emulsion could be uniformly distributed throughout the pulp mixture without causing aggregation or other problems that could interfere with the molding procedure. In order to maintain its uniformity, water was added to the emulsion at an exact ratio and mixed gently. The careful oversight of this process was conducted to prevent the formation of large droplets or clusters, which may result in inconsistent distribution and possible defects in the final products. All chemicals were added to the low consistency, 0.5%, dilution tank with a 3-min low shear mixing time between additions. A circulation pump at the bottom outlet valve of the holding tank before the pulp slurry tank of the thermoformer machine was used to circulate the slurry to ensure that the PSP stayed well distributed in the pulp slurry prior to delivery to the thermoformer machine. The chemically treated pulp was consumed within 1 h of the chemical additions. Molded pulp food trays with dimensions of 240 × 185 × 43 mm (length × width × depth) were produced.

Two different grinding methods were used to produce the PSP, an impact mill and a cryo-ground method. The impact mill ground PSP was evaluated at 5, 7.5, 10, 12.5, 15, 20, and 25% by weight on the DLK pulp. Afterwards the cryo-ground PS powder was evaluated at 10, 20, 25, and 30% by weight on DLK pulp. Table 2 describes the different food tray samples produced.

Table 2. Number of Available Trays by Percent of Added PS Powder and Grinding Method

| PS Powder (%) | Number of Trays | Grinding Method | Group Description |
|---------------|-----------------|-----------------|-------------------|
| 0 | 3 | None | Control |
| 5 | 9 | Impact mill | I-5 |
| 7.50 | 9 | Impact mill | I-7 |
| 10 | 9 | Impact mill | I-10 |
| 15 | 9 | Impact mill | I-15 |
| 20 | 9 | Impact mill | I-20 |
| 25 | 3 | Impact mill | I-25 |
| 10 | 4 | Cryo-ground | C-10 |
| 20 | 4 | Cryo-ground | C-20 |
| 30 | 4 | Cryo-ground | C-30 |

The thermoforming process parameters are presented in Table 3.

Table 3. Thermoforming Process Parameters

| Parameter | Type/Amount | Parameter | Type/Amount |
|-----------------------------|----------------------------------|---------------------------------------|-----------------------|
| Thermoforming Type | Type 3, Thinwall | Forming Pressure | 70 kg/cm ² |
| Number of Stations | 1-Forming, 2-Heating/Pressing | Compressed Air Pressure (System) | 6 bar |
| Pulping Time | 35 min (High Shear) | Forming and Heating Mold Vacuum | 65 mmHg |
| Pulping Consistency | 4% | Mold Press Time | 13 s |
| Freeness | 553 mL CSF | Time to Produce a Finished Product | 38 s |
| Chemical Addition | Low Consistency Tank, 0.45% | Finished Product Moisture | 3% |
| Consistency at Thermoformer | 0.45% | Finished Product Weight | 39 g |
| Mold Temperature | 180 °C | - | - |

The main goal in this work was to provide a sustainable and eco-friendly packaging solution. It was decided to eliminate formaldehyde-containing resins, considering the growing public and legislative attention on lowering the use of dangerous chemicals in food packaging. Less favorable for uses in food-related goods, formaldehyde is well-known for its possible health hazards and categorization as a likely human pollution. Furthermore, the aim was to develop a binder-free product using the self-bonding qualities of the papermaking fibers. During the molding process, papermaking fibers spontaneously showed hydrogen bonding, which may provide enough mechanical strength without the requirement of further synthetic binders (Małachowska *et al.* 2021). This method not only streamlines the production process, but it also fits the goal of generating safer and environmentally friendly goods. The expected mechanical qualities were achieved without sacrificing health, safety, or environmental impact by focusing on the intrinsic bonding capacity of the fibers and the careful use of additives like PSP. Thus, the choice to eliminate formaldehyde-containing resins was both a reasonable attempt to preserve the sustainability of the product and a precaution to guarantee it is appropriate for use in food packaging applications.

Specimen Preparation and Mechanical Testing

Three trays of each condition (Control, Impact mill with 20 wt% PS, and Cryo-ground with 20 wt% PS) were used to conduct the non-destructive tray compression tests. The bottoms of the trays were separated from the rim and cut into clear and flat specimens for mechanical and barrier testing, as shown in Fig. 2(a). All specimens were conditioned and tested in a standard atmosphere according to the TAPPI T402 sp-08 standard (2008).

The thickness and basis weight of the specimen were determined according to TAPPI T411 om-97 standard (1997) and TAPPI T410 om-08 standard (2008) respectively. The tensile strength of the specimens was measured according to TAPPI T494 om-96 standard (1996), in a tensile tester with a gap of 50 mm. The tensile strength, stretch, tensile energy absorption (TEA), and stiffness were measured. The tensile index was calculated by dividing the tensile strength by the basis weight. The bending resistance (stiffness) was

determined according to the TAPPI T489 om-08 standard (2008). The average and standard deviations of bending moment and resistance to bending were reported.

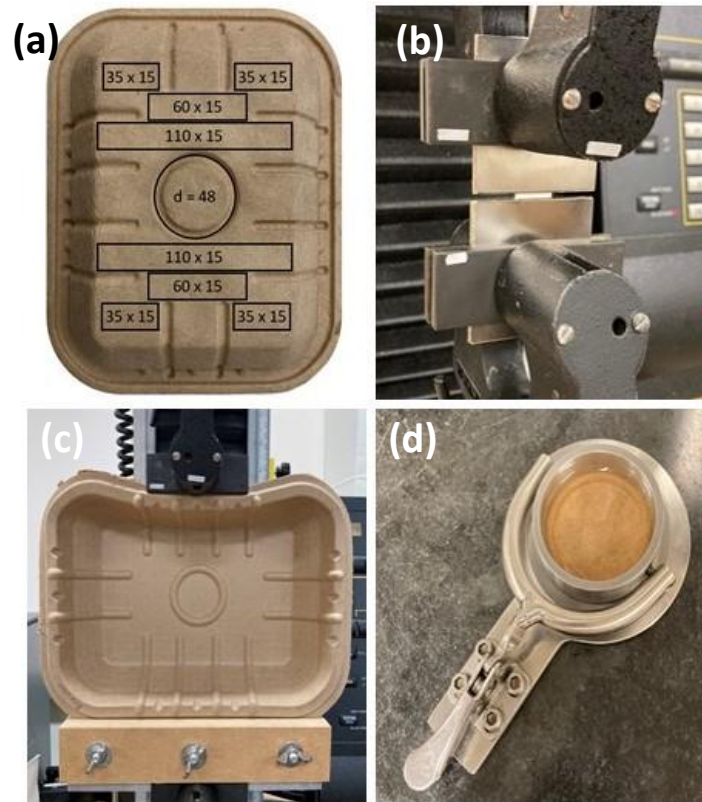


Fig. 2. (a) Scheme of sample preparation for mechanical and barrier testing of pulp molded trays (all measurements are given in mm), (b) Short-span compressive strength testing with Instron, (c) Compression test of pulp molded trays, simulating the compression due to plastic wrap, (d) Water absorption tester with 46 mm diameter using 10 mL of water

The short span compressive strength was determined in accordance with the TAPPI T826 om-08 standard (2008), using a Universal Testing System (Instron / 4443). Due to the thickness of the material, a standard device for short span compression, as required by TAPPI T826 om-08 standard (2008), could not be used. A clamping device was produced from 1-mm-thick stainless-steel sheet, Fig. 2(b). Squares of $50 \times 50 \text{ mm}^2$ were coated on one side with sandpaper of grit 220, to avoid slipping of the specimen during the compression stage. The specimens were clamped between two of the squares on each end, leaving a 2-mm gap (Fig. 2(b)). The measurement was conducted with a 0.5 kN cell at a speed of 200 mm/min and a maximum displacement of 1.5 mm. Twelve specimens per condition were tested. The short-span compressive strength was calculated by dividing the maximum force recorded during the first millimeter of displacement, by the specimen width in meters. The average and standard deviations were reported for each condition.

The tray's compressive strength was measured by compressing along the shorter dimension (185 mm) in a Universal Testing System (Instron / 4443) equipped with a 0.5 kN cell, Fig. 2(c). The bottom edge of the trays was clamped at full length and kept stationary during testing, while the top edge was clamped, centered with a metal clamp at

a width of 78 mm (Fig. 2(c)). The upper metal clamp was moved downwards at a speed of 2.5 mm/min up to a maximum deflection of 18.5 mm, constituting 10% of the tray width.

The Cobb test for water absorptiveness was conducted according to the TAPPI T411 om-98 standard (1998), using a modified water absorption tester with a diameter of 46 mm (Fig. 2(d)). Because the surface of the specimens was not entirely flat, but showed an elevated rim, the diameter for calculating the surface area was corrected to 48 mm. The specimens were exposed to 10 mL of distilled water for 2 min. Water absorption was calculated by subtracting the dry specimen mass from the specimen mass after water exposure. The absorptiveness was calculated by dividing the mass of water absorption (g) by surface area (m²). Three repetitions were conducted for each condition, and the average and standard deviations were reported.

Characterization Tests

For performing SEM-EDS, the samples were mounted on an aluminum stub by attaching to a sticky carbon surface above the aluminum stubs. The mounted samples were sputter coated with a thin layer of gold material to make the sample conductive to electrons using an EMS150R S sputter coater (Electron Microscopy Sciences, USA). A S4700 II cFEG Scanning Electron Microscope (Hitachi High Technologies, USA) with a silicon drift EDS detector (Oxford Instruments, X-MaxN, UK) was used to measure the surface morphology, elemental composition, and distribution of elements. All the SEM data reported were obtained at an acceleration voltage of 10 kV, and the images were collected with a secondary electron detector. The elemental mapping and energy spectrums were acquired with the Aztec tools (Oxford Instruments, UK).

The particle size distribution (PSD) of the PSP was measured *via* the laser diffraction method with a Malvern Mastersizer 2000 with Hydro 2000S disperser that was maintained with periodic checks on NIST-traceable polystyrene control samples. The powder samples were dispersed by wetting with ~4 mL 91% isopropyl alcohol (IPA), mixing 2 mL of the slurry in 50 mL of a weak solution of sodium lauryl sulfate, and sonicating with an ultrasonic horn for 60 s. The Mastersizer was filled with deionized water circulating at a pump speed of 1995 rpm. After recording the background diffraction pattern with clean water, about 2 mL of the sample dispersion was added to the Mastersizer. The Fraunhofer approximation was used in the scattering calculations because the refractive index of the ground shell was not known, but the relatively large size of the particles indicated that the approximation was valid.

The XRD spectra were collected by a fine-focus Mo-sealed tube running at 50 kV and 35 mA (Mo K α = 0.711 Å) on a Bruker D8 Venture Diffractometer equipped with Helios multilayer optics, and Burker Photon III C14 detector. The sample preparation included mixing the powder with Paratone N oil and placing it in a < 0.5 mm nylon loop, mounted on a goniometer head. A Bruker APEX4 software package was used to collect and merge 120°. 360° ϕ -scans with the detector at $2\theta = -25^\circ$ and 30° using a sample-to-detector distance of 70 mm. The spectra were collected using an FTIR (Shimadzu IRSpirit) with a diamond crystal ATR accessory (Shimadzu QATR). The samples were analyzed as powders placed directly on the ATR crystal. A total of 16 scans over a range of 500 to 4000 cm⁻¹ were averaged in each spectrum.

RESULTS AND DISCUSSION

Characterization tests of the starting materials and tray products included laser diffraction, SEM, EDS, elemental mapping, XRD, FTIR, and CHO tests. The produced trays were tested for physical properties including basis weight, thickness, density, and cobb tests. The trays were also mechanically tested with tensile tests, bending tests, and short span compression and tray compression tests. The characterization tests, physical tests, and mechanical tests are described below in separate sections.

Characterization Tests Results

Laser diffraction was used to quantify the PSP's particle size distribution; the average size (D50) of the particles generated by the impact milling technique was found to be around 50 μm , with a more general distribution including both smaller and bigger particles. Conversely, the cryo-milled particles produced a smaller size dispersion, as their average size (D50) was closer to 30 μm . Figure 3 (a through d) illustrates the irregular flaky morphology of the PSP powder. The SEM image clearly shows the compactness of the fiber bindings in pulp molded food tray (Control). On both the control and C-20 surfaces, the cross-sections appeared smooth due to compression. I-25 was characterized by a rough and hairy surface. Regarding the particle shape, the impact-milled PSP usually displayed an erratic, flaky form, as indicated by the micrographs. These particles had irregular edges and varied in size, contributing to a more heterogeneous mixture when combined with the pulp matrix. In contrast, the cryo-milled particles were more spherical in shape, with smoother surfaces, which possibly enabled their more uniform distribution within the fiber network. Based on the results of the elemental mapping, and due to the organic nature of the biomass, the elements C and O were only found in the elemental composition of PS powder (Fig. 3(e)). Elemental mapping shows that the particle size distribution of C, O, Si, Al, and Ca elements is uniformly distributed in the pulp molded trays (Figs. 3(g through h)).

Figure 4(a) shows the FTIR spectra of PS powder, pulp molded food tray (Control), I-25, and C-20. The FTIR spectra at 600 to 900 cm^{-1} correspond to vibrations of C–H. In the area of 1030 cm^{-1} , there were intense bands associated with O–H vibrations in cellulose (Konsolakis *et al.* 2015). Cellulose exhibits the scissoring motion of CH_2 in an absorption band of 1430 cm^{-1} . The peaks at 1263, 1507, and 1746 cm^{-1} are attributed to the C–O–C stretching in lignin and cellulose, C=C bonds in aromatic rings in lignin, and stretching of C=O bonds in carbonyl, ester, and acetyl groups in xylan. The band at 1601 cm^{-1} corresponds to aromatic C=O rings and aromatic or C=O stretching groups in lignin. A peak at 2904 cm^{-1} is attributed to the aliphatic C–H vibrations. There is also evidence of hydroxyl group (–OH) stretching in the broad peaks at around 3355 cm^{-1} . There was no significant difference observed for the different levels of PSP added.

As shown in Fig. 4(b), XRD analysis was performed on the PSP powder, pulp molded food tray (Control), I-25, and C-20. The XRD pattern with peaks at $2\theta = 22.8^\circ$ is attributed to the (002) reflection plane specific to the lignocellulose framework (JCPDS card no 03-0289) and is in agreement with the samples containing lignocellulose, specifically cellulose (Cottayil *et al.* 2015; Kumar *et al.* 2021). The broad peak at 16.1° is likely due to the overlap of the (110) plane, which is consistent with the diffraction pattern of cellulose. In this diagram, the peak at $2\theta = 34.5^\circ$ is assigned to the (004) plane (Wan *et*

al. 2019). The laser diffraction result for PS powder (ground or cryo crushed) is shown in Fig. 4(c). The median size of the sample is close to 626 μm ; in this case, 10% of the volume belongs to the particles smaller than 14 μm , whereas 90% of the volume belongs to the particles smaller than 635 μm .

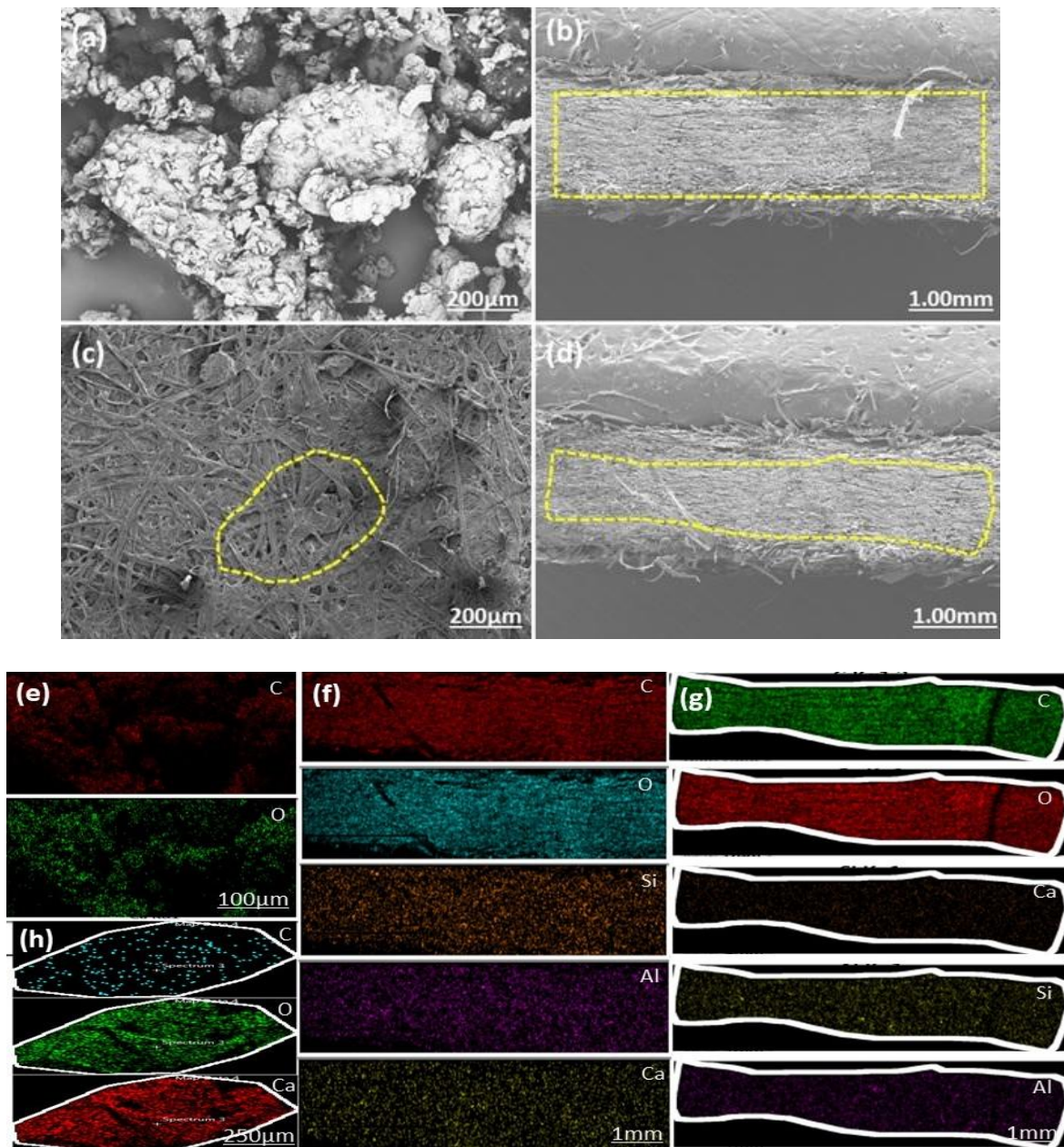


Fig. 3. (a) SEM images of PS powder, (b) pulp molded food tray cross-section (Control), (c) I-25 tray top surface, (d) C-20 tray cross-section, (e) Elemental mappings of PS powder, (f) pulp molded food tray (Control), (g) I-25, and (h) C-20

The EDS analysis of the pulp molded food tray (Control) showed peaks for C, O, Si, Al, and Ca (Fig. 4(e)). The EDS spectrum analysis of pulp molded tray (I-25) contained peaks for C and O. The EDS spectrums, shown in Fig. 4(e through g), indicate the presence

of elements, such as Ca, Si, and Al, which are considered impurities in the recycled fiber (DLK). According to these results, I-25 and C-20 had been homogeneously blended into the fiber network of PS powder and become intimately in contact with the fibers. Therefore, this issue further indicates that fiber-fiber interactions and bonding within blended systems had increased. Because the PSP is lignocellulosic and can participate in hydrogen bonding with the fibers, it is possible that the PSP could contribute to physical and mechanical properties, as discussed below.

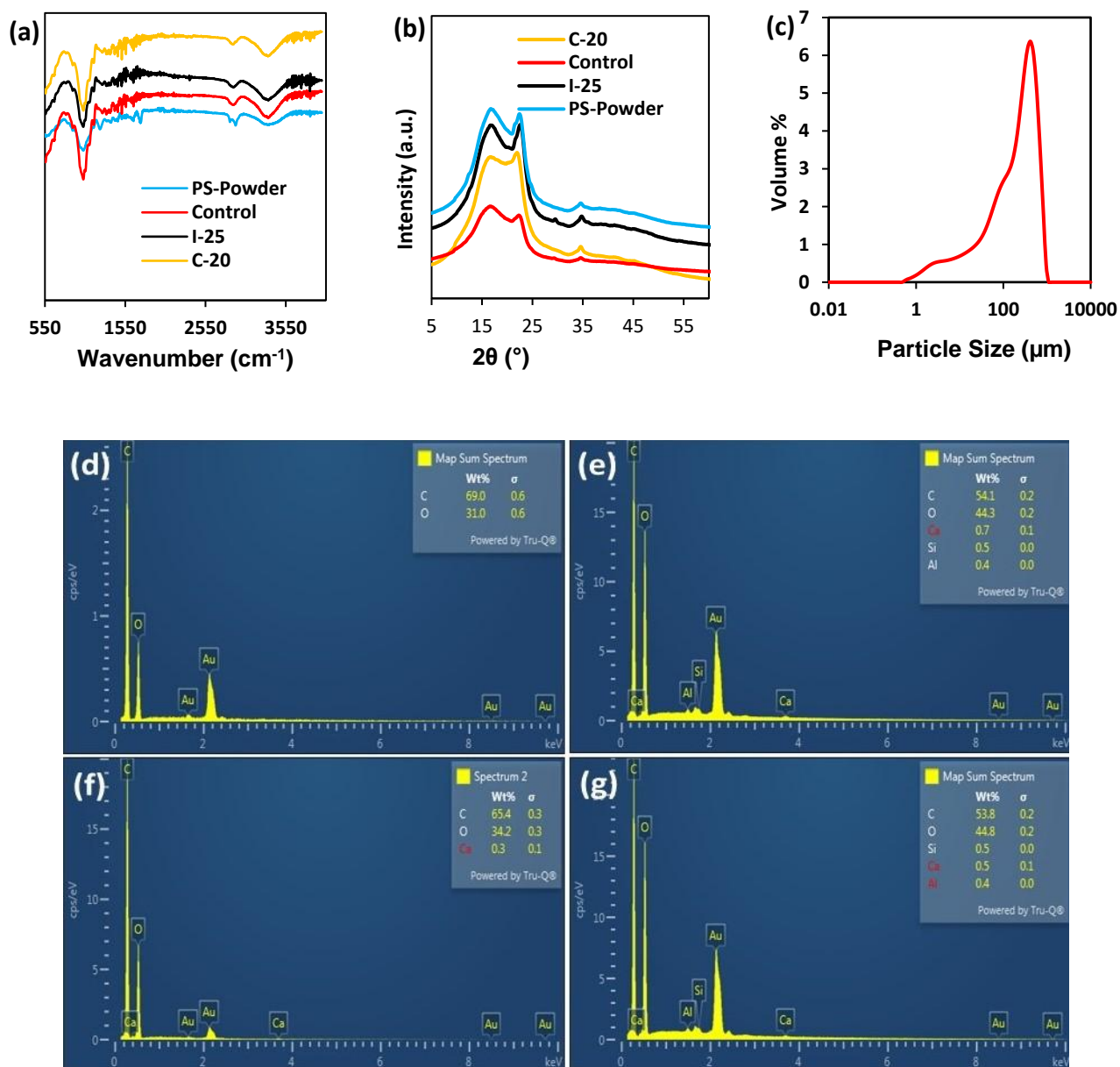


Fig. 4. (a) FTIR, (b) XRD of PS powder, pulp molded food tray (Control), I-25, and C-20, (c) Analysis of particle size distribution via laser diffraction of PS powder, EDS analysis of (d) PS powder, (e) pulp molded food tray (Control), (f) I-25, (g) C-20

The chemical composition of PS powder contained glucan 27.7 ± 0.5 , xylan 28.1 ± 0.4 , arabinan 1.0 ± 0.1 , LK (Klason Lignin) 24.6 ± 0.2 , moisture 4.1 ± 0.2 , fat 2.5 ± 0.0 , protein 3.8 ± 0.0 , and ash 1.1 ± 0.2 . The CHO analysis was used to identify the elements in the samples, Table 4. It is observed that the PSP had higher carbon and lower oxygen than the DLK pulp in the control. However, these differences were not very significant and the results between the control and the trays with PSP did not discern a significant difference with respect to the measurement capabilities of the test.

Table 4. Percentages of Carbon, Hydrogen, and Oxygen Present in PS Powder, Pulp Molded Food Tray (Control), I-25, and C-20

| Sample | Carbon (%) | Hydrogen (%) | Oxygen (%) |
|-----------|------------|--------------|------------|
| PS powder | 48.58 | 6.99 | 41.96 |
| Control | 45.49 | 5.76 | 44.01 |
| I-25 | 45.59 | 5.71 | 44.26 |
| C-20 | 45.10 | 6.10 | 44.34 |

Physical Tests Results

All trays were manufactured with the same operating parameters. There were no runnability issues on the industrial pulp molding equipment. The production rate of the PSP containing trays, often limited by the drying process, was indistinguishable from the control without PSP. No unusual deposits were found on the machine forming screen or pressing and drying plates. The process water that was recirculated in the system was of normal condition with respect to suspended solids over almost 8 h of production. The I-20 had the highest basis weight, approximately 609 g/m^2 , among all the molded trays, which weighed between 514 and 560 g/m^2 . The thickness of the specimen was determined with a caliper (Lorentzen & Wettre / Micrometer 51). The I-20 and C-20 were $852 \mu\text{m}$ and $840 \mu\text{m}$ thick, respectively. While the density of the control tray was 630 kg/m^3 , after blending with PS powder, the density of I-25 and C-20 increased to 715 and 655 kg/m^3 , respectively (Fig. 5(a through c)). This suggests that the PSP may be filling voids in the fiber matrix, which increases the density of the structure.

Another important factor for a food tray is the resistance to water/liquid absorption. The results of water absorptiveness are depicted in Fig. 5(d). The absorptiveness, as measured with a Cobb test of the Control, I-20, and C-20, was 24.1 , 27.3 , and 37.4 g/m^2 , respectively. The I-20 sample absorption was similar to the control whereas the C-20 shows a 55.3% increase in the absorption. The results correlated with the inverse of the density of I-20 and C-20, with lower density materials presumably having a more porous structure and absorbing more water.

A comparison of the tensile index for pulp molded food trays of control, I-20, and C-20 is shown in Fig. 6. The tensile index of C-20 was lower (34.3 Nm/g) than those of I-20 and control (38.8 s and 39.4 Nm/g , respectively). In the control tray, the fibers had a high content of lignocellulosic fibers (structural component), resulting in high fiber-fiber interactions and bonding. There was more flexibility in the control tray and a larger contact area for fiber bonds as a result (Gulsoy and Tufek 2013; Saeed *et al.* 2017; Rattanawongkun *et al.* 2020). The impact-milled sample (I-20) showed a similar tensile test, with an 11.3% increase compared to the control sample. The tensile properties of the PSP containing pulp molded food trays were in line with the tensile index of the control, which represents

commercially viable molded pulp packaging (Fig. 6). Of the samples, C-20 had the lowest density, indicating lower bonding, in agreement with the lower tensile index.

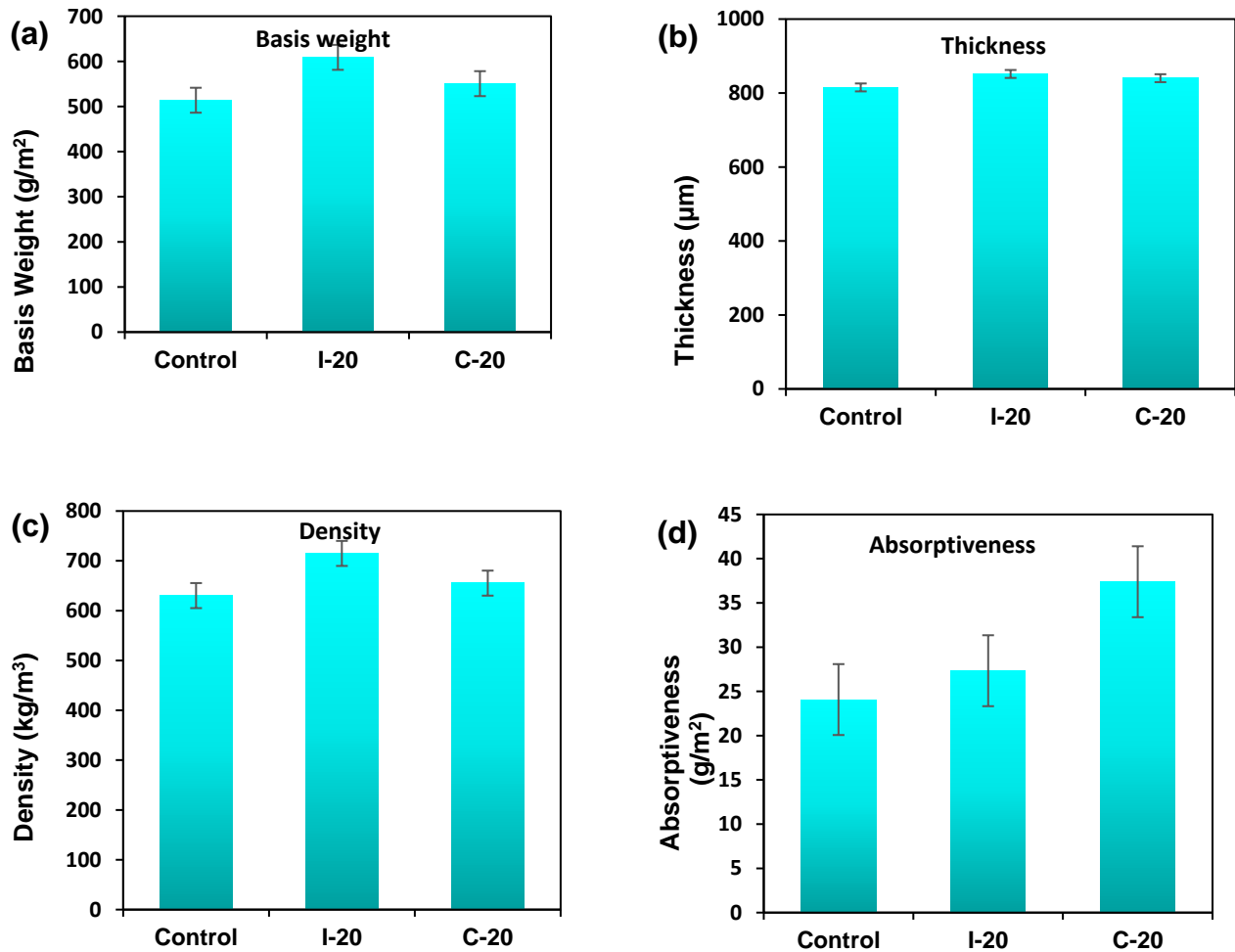


Fig. 5. (a) Basis weight, (b) thickness, (c) density, and (d) water absorptiveness (Cobb test) of pulp molded food tray (Control), I-20, and C-20

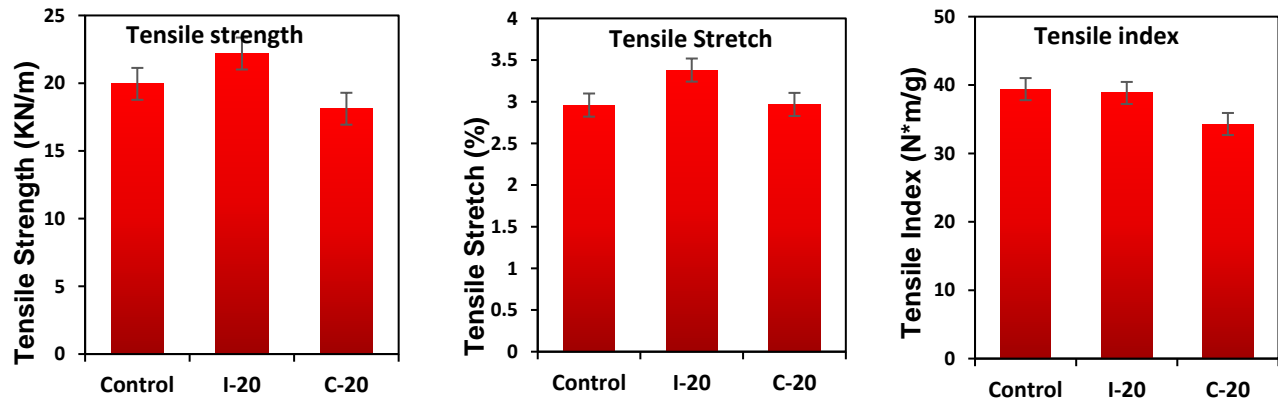


Fig. 6. Tensile strength, stretch, and tensile index of pulp molded food tray (Control), I-20, and C-20

Bending resistance is one of the most important properties of a food tray. The bending moments for the Control, I-20, and C-20 samples were 16.6, 17.7, and 16.4 mN.m, respectively. The bending resistance for the Control, I-20, and C-20 samples were 327.4, 353.2, 328.4 mN, respectively. Sample I-20 had a bending moment and resistance 6 and 8% higher than the control, respectively. It is of interest to note that the bending resistance was correlated to the density of the materials in the same manner that the tensile index was, reflecting more fiber bonding with higher density materials.

Short-span compressive strength of the walls is important in food trays to resist crushing under the loads of stacking filled trays. An example of the force's development as a function of displacement in the short-span compression test is presented in Fig. 7(c).

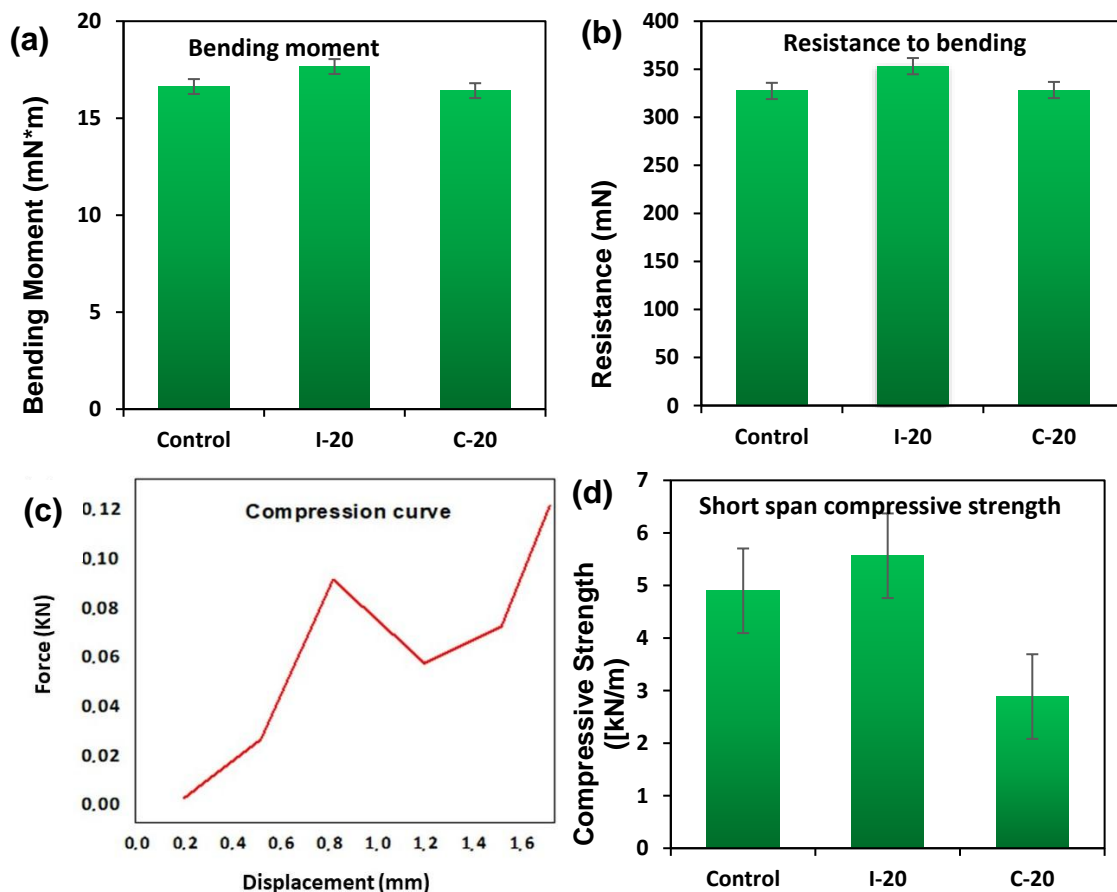


Fig. 7. (a, b) Bending moment, and resistance to bending of pulp molded food tray (Control), I-20, and C-20, (c) Example short span compression curve as measured during the short span compression test, and (d) Results for short span compressive strength

First, the maximum force before material failure was reached at 0.6 to 0.8 mm displacement, followed by a decrease in force with increasing displacement up to 1 mm. Eventually, the force increased while the specimen, at a thickness of around 0.9 mm, was compressed in the gap of < 1 mm between the clamps, an artifact of the test procedure. The maximum force at displacement < 1 mm was considered as a reasonable measure for evaluating the short-span compressive strength. According to Fig. 7(d), the short-span compressive strength for the Control, I-20, and C-20 samples were 4.90, 5.56, and 2.89

kN/m, respectively. I-20 showed the highest magnitude, with a 13.5% increase compared to the Control. The C-20 result was lower than the Control, again in line with increased density and bonding of the samples.

These results suggest that the overall mechanical properties may indicate more interference in fiber bonding with cryo-ground PSP compared to impact-milled PSP. Current research with the PSP material incorporated into fiber-based containerboard is ongoing, better defining the extent of retention and locations of the PSP in the fiber matrix as a function of retention aid aqueous-based polymer system addition. The ongoing research should be useful to define efficient conditions in which to incorporate PSP in fiber-based products. Overall, this study demonstrated a method to evaluate pulp molded products with respect to the addition of additives as well as showing that PSP could be added to pulp molded food trays resulting in similar and reasonable product properties. This product application for PSP demonstrates a reasonable method to re-use PSP, addressing PSP end of life concerns.

It is worth noting that conventional paper tests, such as those for tensile strength and bending resistance, are meant for flat specimens. To evaluate the mechanical qualities of the non-flat, molded pulp products of the present study, the approach was to modify these conventional testing techniques. This method made it possible to apply standardized testing to the molded pulp products in a manner that would be comparable to other materials and research efforts. Testing flat pieces made it possible to get important mechanical property data including tensile strength, bending resistance, and other mechanical characteristics, which are qualities that are absolutely essential for assessing the trays' general performance. Although the trays themselves are three-dimensional and non-flat, the flat sections that were examined showed a consistent behavior of the material under usual stress situations. Commonly utilized in the research projects of molded pulp products, this process offers dependability in the comparison of findings across many studies and materials. This method allows a good evaluation of their mechanical qualities and would essentially reconcile the need for standardized testing with the special qualities of molded pulp products.

Pulp Molded Tray Compression Tests

Trays were evaluated for their resistance to compression forces perpendicular to their long edge. This is a critical final product attribute because the trays are normally wrapped with a protective plastic film by automated and rapid packaging machines to seal the product completely. This creates forces and deformations as normally observed in practice. This was demonstrated with a finite element analysis (FEA) model using the analysis tool ANSYS Workbench 2020 R1 during the design phase and prior to manufacturing the prototype by forming molds. The 3D mesh structural build with approximately 190,000 elements and 465,000 nodes was created with the following material properties based on previous designs for a one-to-one comparison:

Density: 0.95 g/cm³, Modulus: 5,000 MPa, and Poisson's ration: 0.4

In the model, a 10 lbf (0.045 kN) downward force on the long side of the tray was imposed with other parallel edges in a fixed support. The results of the model showed a maximum total deflection of 0.108" (2.74 mm) with 10 lbf (Fig. 8).

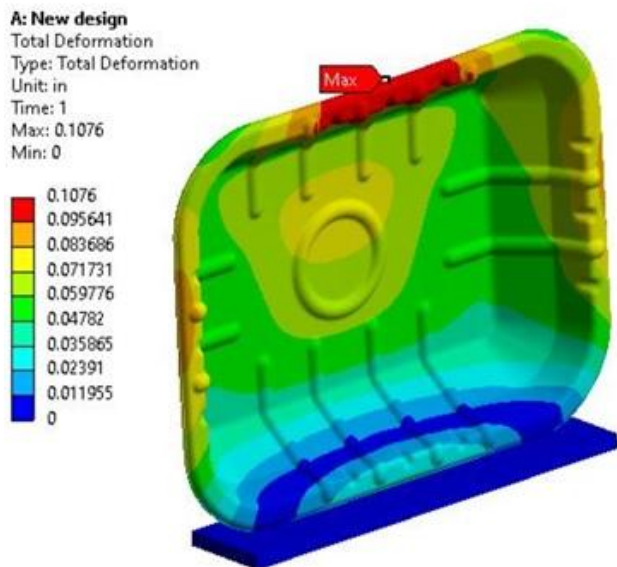


Fig. 8. FEA analysis of tray (ANSYS Workbench 2020 R1)

Distinctive Properties and Potential Advantages

These studies mainly sought to find out whether PSP may be a suitable filler for molded pulp products without compromising mechanical and barrier qualities. It was shown that PSP may replace some of the papermaking pulp while preserving these characteristics within a suitable range. This result is important, as it shows that a plentiful agricultural waste product might be used efficiently in commercial packaging uses, and lowering the need on virgin pulp resources. It is important to find out typical properties that could set these products apart from the traditional molded pulp products. Comparatively to the control trays, the trays with PSP – especially those constructed with impact-milled particles – had a rougher surface. Given the uneven and flaky character of the PS particles, this roughness might provide a more natural, organic feel that would be appealing in certain packaging uses. The addition of PSP also changed the trays' density and porosity. For example, trays constructed from impact-milled powder had lower porosity and higher density; therefore, changing the barrier characteristics and interaction with other materials. Although the friction coefficient was not investigated in great depth in this study, the rougher surface and higher density of the trays containing PSP suggest that this quality might differ from ordinary molded pulp products. This might influence the handling and stacking of trays in industrial settings; thus, it is proposed that future research focus on this aspect.

CONCLUSIONS

Nutshells have great potential for use in composites and fiber-based molded parts. Among the nutshells, pistachio shells make up approximately 100,000 tons per year of waste in the U.S. These waste shells have been shown in this research to have the potential to be added to pulp molded products in a commercial manufacturing system. Minimal

modifications to the commercial process were required to add up to 30 wt% pistachio powder with normal runnability of the machine. Major findings include:

1. The pistachio shell powder (PSP) materials could be added to pulp molded products in an industrial manufacturing line without compromising machine runnability or product properties.
2. The X-ray diffraction (XRD), Fourier transform infrared (FTIR), and elemental (CHNO) analysis confirmed that the PSP material had similar characteristics to other lignocellulosic and therefore could be expected to have potential to be incorporated into and bond with lignocellulosic fibers.
3. The scanning electron microscopy (SEM) image exhibited the compactness of the fiber material in the pulp molded food trays and showed evidence of the PSP incorporated into the composite structure.
4. It was observed that the addition of PSP could impact the density of the fiber-based matrix and that this positively correlated with mechanical properties, such as tensile, bending, and short span compression, of the pulp molded tray wall material, which was attributed to higher bonding of the fiber based matrix in the denser materials.
5. Pulp molded trays produced using PSP made with the impact mill showed improved behavior relative to the control, whereas the cryo-crushed PSP had similar or slightly lower properties relative to the control. The origin of these differences should be investigated in future research.
6. The compression resistance of the overall tray perpendicular to the long edge of the tray is critical; PSP containing trays showed similar compression resistance as the control for the entire tray in this direction.
7. The methods developed in this work can be used as a guideline for testing future pulp molded materials, especially regarding additional components such as alternative fiber sources or other additives.

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