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Feasibility of using straw in a strong, thin, pulp moulded packaging material.

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Abstract

Packaging is a ubiquitous commodity that is being used in increasing quantities. This increased use has led to a problem with disposal, with increased quantities of used packaging being sent to landfill. One sustainable solution suggested is the use of biobased, biodegradable packaging. An example of this is paper based pulp moulded products which have been used previously for a number of packaging applications. In this paper the feasibility of replacing paper fibre with waste cereal straw fibre is examined. The aim was to produce materials that could be used to form flat, round trays, such as

those used in supporting shrink wrapped food items. The material was required to have properties that matched existing alternatives, such as expanded polystyrene, in terms of physical and mechanical characteristics but with an enhanced level of biodegradability. The data showed that the pulp moulded material containing up to 80% straw performed significantly better compared to expanded polystyrene in tensile properties (modulus of 0.47 MPa for an 80% straw mix compared to 0.16 MPa for EPS). Modulus under bending was shown to be lower for straw based materials compared to EPS (0.015 MPa compare to 0.035MPa). Adjustments in product thickness allowed performance parameters to be met. Wet end addition of chemicals was successfully used to provide water resistance without affecting other variables. In addition to exhibiting good performance characteristics the pulp moulded material was shown to be biodegradable, exhibiting 20% mass loss after only 4 weeks covered in unsterile soil.

Highlights

- Pulp moulded flat, round packaging trays were produced using straw.
- Intrinsic tensile properties improved compared to Expanded Polystyrene (EPS)
- Intrinsic flexural properties of straw material were lower than EPS.
- Product performance matched EPS performance with changes to product thickness
- Straw based materials were biodegradable.

Key words: Pulp moulding, straw, packaging, renewable, biodegradable

1.0 Introduction

Single use packaging materials are a ubiquitous feature of modern society and, in the United Kingdom, have shown rapid increase in their use. However, with recent legislative pressures, such as those described in the European Union Landfill directive, [European Union 1999] and other societal consumer concerns [Hall et al 2010], alternative biobased solutions to petrochemical based plastic packaging are being sought. In terms of managing waste at the end of life of the product, enhanced biodegradability of biobased packaging is an essential asset [Siracusa et al 2008, Song et al 2009] as many packaging materials are not reused but thrown into waste – with these non biodegradable products adding to the land fill burden. A key attribute of a biobased packaging product should, therefore, be an improvement in biodegradability over existing alternatives. However, biodegradable items can cause contamination in plastic recycling and ideally therefore, the whole product should be biodegradable at the level of either a community or household composting regime [Davis and Song (2006)]. One currently used form of biobased packaging moulded paper pulp packaging [Guray et al 2003; Zabaniotou and Kassidi 2015], where the packaging is mainly used for its cushioning properties [Eagleton and Marcondes 1994; Hoffmann, 2000]. Other studies [Gurav et al 2003,] have investigated the strength properties of biobased moulded packaging created via wet forming. Wet forming uses a water borne pulp fibre suspension shaped in a mould with simultaneous or subsequent (based on the equipment used) dewatering and drying. Wet formed packaging material of this type has mainly used recycled paper and cardboard fibre, although in some cases, such as food contact, this is not deemed viable due to contamination from inks *etc.* in the pulp. An alternative to waste paper would be pure Kraft pulp but this may not be economically viable. However, other non-wood based lignocellulose feedstocks, such as cereal straw, could be used to produce the pulp. Each year approximately 1.45Mt of cereal straw [Glithero 2013] are reincorporated into arable soil. This is partly as a waste management issue although the straw does also serve as a soil amendment and improver. On availability grounds,

straw can therefore be considered a good candidate as a raw material in biobased materials. In terms of packaging, straw based packaging applications have been developed [Vargas et al 2012] although, to the author's knowledge, the particular use of straw in the manufacture of thin sheet materials, does not appear to have been reported. These thin sheet materials, commonly made from expanded polystyrene are frequently used to help support products in food applications, such as the round trays used in packaging to support cakes, pastries or pizzas to stop bending of items during shrink wrapping and display. The current study is part of a larger project to develop a flat, round straw based tray that could be used for the applications noted above.

In terms of moving a biobased product to commercial viability the product must perform mechanically at least as well as an established alternative but also be biodegradable. This paper therefore investigates the feasibility of using straw pulp to produce material suitable to form a pulp moulded, flat, round tray by testing the materials' and products' mechanical and biodegradation properties. Expanded polystyrene products are used as a comparative benchmark for required properties.

2.0 Materials and methods

2.1 Preparation and Characterisation of raw materials.

Wheat straw (*Triticum aestivum* cv Solstice) was selected as the straw component. The straw was cut into pieces smaller than 5cm and then refined using a 30cm pressurised refiner (Andritz Sprout Bauer). Briefly, this refining process consisted of the followingn steps;

The straw was fed via a cooker screw into a 60 litre digester, with a nominal residence time of 60 seconds in the cooker screw and digester. Steam pressure in the cooker/digester was maintained at 0.94 MPa using steam at 390°C. On leaving the cooker/digester, the straw was fed, via a second screw feed, into the refiner, and passed between two 30cm diameter refiner

plates, with a parallel bar configuration, to form the fibre (fuller details can be found in Ormondroyd et al 2016).

The starting straw and resultant pulp pre-mix were chemically characterised as follows:

2.1.1 Wax/Extractives: Replicate samples were extracted with toluene, acetone, and methanol (4:1:1) for 8 hrs using soxhlet extraction (at least 50 solvent cycles). Wax/extractive content were calculated and expressed as percentage on a dry weight basis.

2.1.2 Klason Lignin: Tappi standard method 222 [Tappi 2006] was used to determine lignin concentration in the samples. This method used acid hydrolysis of the polysaccharides in 72% sulphuric acid leaving the lignin in solid form. Lignin content was expressed as percentage on dry weight basis.

2.1.3 Alpha cellulose and Hemicellulose: The alpha cellulose and hemicellulose contents of the samples were determined by analysis of holocellulose isolated using the sodium chlorite method [Browning 1967] This used acidified sodium chlorite to delignify the samples leaving holocellulose, which was extracted using 17% sodium hydroxide. Following neutralisation with acetic acid and washing with water and methylated spirit, the alpha cellulose was separated by filtration. Composition was determined gravimetrically as a percentage of original dry weight. Hemicellulose content was determined by neutralisation of the filtrate and precipitation of hemicellulose via addition of copious ethanol. Following drying of the hemicelluloses the composition was determined gravimetrically as a percentage based on original dry weight of sample.

2.1.4 Ash: Oven-dried material was ashed in a muffle furnace at 525°C for 16 hrs, with the ash content calculated as a percentage on dry weight basis.

2.2 Pulp moulding. The pulp for the moulding of the material was prepared in a mixing tank of a proprietary pulp moulder (Valueform, UK) utilising a plain transfer moulding process. Kraft paper pulp fibre was obtained from commercial dried sheets which were pulped in water before adding to the straw fibre pulp at three differing ratios – a base 100% straw mix, a 80/20 straw/Kraft mix and a 60/40 straw/Kraft mix. To these mixes an anti-foaming agent (Percol, BASF) and a water repellent additive (Basoplast, BASF) were added. The water repellent additive was included to decrease the water absorbance of the final product, although some samples without added water repellent were produced to test the efficacy of the additive. The anti-foaming agent was added to stop a foam or froth forming in the pulp as this was found to cause holes in the product during the moulding cycle. Although the exact details of the moulding cycle are not reported for potential commercial reasons, the procedure consisted of immersion of the mould into the pulp, filling of the mould, vacuum removal of water and pressing of the pulp using proprietary cycling schedules. The moulded material was dried by hot air convection in an oven at 100°C for 30 minutes.

2.3 Characterisation of pulp

The pH, drainage and particle size distribution of the pulp were measured prior to moulding. The drainage of the pulp was assessed using Canadian Standard Freeness of the pulp following the method described in ISO 5267-2 [ISO 2001]. Particle size distribution was determined by drying the pulp to a fibrous state, with no agglomerations, and then sieving through a sequential series of wire mesh sieves with frequency allocation based on mass fraction.

149 *2.4 Characterisation of moulded material*

150 In the investigation detailed in this paper the 100% straw mix did not produce a moulded
151 product that was suitable for use or analysis. Therefore, only samples representing the 80/20
152 and 60/40 mix were analysed in terms of physical characteristics, mechanical properties (tensile
153 and flexural properties, water absorbency and biodegradation. Due to limitations placed by the
154 dimensions of the product, in this case a round tray, non-standard testing procedures were used
155 in some instances. To validate the study, controls of similar existing products were tested in
156 the same way as the moulded products.

157

158

159 *2.4.1 Physical characterisation*

160 The thickness of the product material was determined using digital callipers and reported as a
161 mean value of at least 5 replicates per sample. The density of the material was determined
162 gravimetrically.

163

164 *2.4.2 Mechanical testing*

165 The tensile properties of the product materials (conditioned at 20°C and 65% relative humidity
166 (RH)) were determined using an Instron testing machine (Instron, High Wycombe, UK) using
167 a 5kN load cell, in a controlled environment (20°C and 65% RH). Samples of size 80mm by
168 25mm by thickness (See Table 2 for thickness) with a gauge length of 50mm were subjected
169 to a uniform rate of tensile force until failure. Samples of EPS of the same dimensions were
170 also tested in the same way. Modulus (equation 1) was determined from the linear portion of
171 the load/deformation relationships.

172 $$modulus = \left(\frac{F}{wd}\right) \Big/ \frac{\Delta}{l} \qquad (1)$$

Where F = force applied (N), w = width (mm), d=depth (mm), Δ= deflection, l = span length

The flexural properties of the product material were tested under 3 point loading also using an Instron testing machine (Instron) using a 5kN load cell, in a controlled environment (20°C and 65% RH). Specimen size was 80mm by 25mm by thickness and the test span length of 50mm. Bending was performed to a set deflection rather than rupture as it was observed that the straw based material did not give an identifiable rupture point during testing. Initial tests were performed to identify the linear portion of the deflection curve prior to the yield point to set this deflection point. Following a small preload a uniform rate of loading was applied until a maximum deflection of 5mm was obtained. The modulus (equation 2) was determined from the resulting linear portion of the deflection graph. Samples of EPS of the same dimensions were also tested in the same way to allow comparative analysis.

$$Modulus\ (bending) = \frac{Fl^3}{4\Delta bd^3} \quad (2)$$

Where F = force applied (N), L= span length, Δ = deflection, b=breadth, d= depth.

2.4.3 Water absorbance

Water absorbance of the product material was determined using the Cobb test [Jacobs et al 2002]. A known volume of water (100ml) was applied to a defined area of the material (100cm^2) and uptake of water was determined gravimetrically after a period of 120 seconds. The water absorbance of samples not treated with the water repellent was also measured to determine the efficacy of the additive.

2.4.4 Biodegradability

The biodegradability of the samples was determined using a soil box method utilising unsterile soil (Li et al 2007, M. Venäläinen et al 2014), Samples (11cm x 3cm x thickness) were cut from the test products. The samples were placed into a soil box where the samples rested on and were covered by unsterile moist (30-35% moisture content) soil (John Innes No 2). The test assemblages were maintained at room temperature for four weeks before assessment by visual and mass loss criteria. This test is a non-standard test developed from methods described by Curling et al (2002) and the standard method ENV807 (British standards 2001).

2.5 Statistical procedures

Where appropriate simple statistical analysis of the data was performed using t-tests assuming equal variances following ANOVA testing.

3.0 Results and Discussion

3.1 Raw material Characterisation

The straw material was analysed for chemical content pre and post refining treatment. The data shown in Figure 1 indicates that the refining changed the composition of the fibre relative to the straw by removing some of the hemicellulose (likely water soluble fractions [Tappi 2015] and ash.

Figure 1 Chemical composition of straw pre and post refining labelled straw and fibre respectively (Error bars show standard deviation).

3.2 Pulp Characterisation

The pulp of the 80/20 and 60/40 mixes were analysed on the basis of pH and Canadian standard freeness and compared to the base refined straw (Table 1). The data shows that addition of the Kraft pulp reduced the Canadian Standard Freeness (CSF) (all differences significant at p=0.05) and increased the pH. The CSF is an important measure when pulp moulding as it indicates the ease at which the moulded product is dewatered – with faster dewatering leading to better integrity and improved drying. Improved drying may be an important factor when considering economic costs of drying the product material.

The determination of the particle size of the mixes in comparison to the base refined straw shows (Figure 2) that the base straw has a majority of shorter size fibres (200 - 400µm) with a general but declining distribution of larger size particles, whilst the 60/40 mix has a skewed distribution towards larger fibres (1680µm and above). The 80/20 mix in contrast shows a distribution of two distinct sizes at 200 - 400µm and 1680µm and above. The large particle size was assigned to the Kraft paper fraction with the smaller size assigned to the straw.

Figure 2. Particle size distribution of pulp mixes.

3.3 Characterisation of moulded material

As stated moulded products produced using 100% straw in this investigation did not produce material suitable for testing or use. It was found during moulding that the product had no wet strength and could not be removed from the mould without breaking. It was assumed that this was due to the lack of the longer Kraft pulp fibres binding the material together. Therefore, the physical characteristics of the moulded material produced from the 80/20 and 60/40 mixes only, is reported. The physical characteristics (Density and thickness) of the moulded material

compared to a commercial expanded polystyrene packaging sheet are shown in table 2. The data shows that the straw based moulded materials had a higher level of variation in thickness (based on standard deviation of thickness). This may have been due to springback of the straw fibres after moulding. However, the 60/40 mix was statistically significantly thinner (at p=0.05) than the EPS whilst the 80/20 mix was statistically significantly thicker (at p=0.05). The data also shows that increasing the level of Kraft pulp increased the density of the material (statistically significant at p=0.05), although it is clear that the straw based materials have a much higher density than the EPS. As the moulded materials had a similar thickness to the EPS but much higher density the weight per unit area of the moulded materials will much higher than the EPS. This may have implications on economics where larger weight transported resultants in a higher cost, although economic metrics were not part of the study detailed in this paper.

3.3.1 Mechanical testing

These tests used nonstandard test sizes making comparison with other studies and materials difficult. Therefore the mechanical properties data has been assessed using two approaches; a) the intrinsic mechanical properties of the material and b), the performance of the moulded product in comparison to the commercial (i.e. EPS) product

The values of the tensile properties of the materials were determined from the linear portion of the load/deformation relationship (representative examples shown in figure 3).

Figure 3. Representative examples of the load/deformation curves for the pulp moulded and EPS samples

The data shows (table 3) that addition of the Kraft pulp significantly increases the tensile modulus, (significant at p=0.05) of the 60/40 mix compared to the 80/20 mix. This may be due to the presence of the larger particles identified by the particle size distribution (Figure 2). In comparison to the EPS both pulp mixes gave modulus values that were significantly higher (at p=0.05) than EPS.

In terms of the flexural properties of the materials, values for modulus under bending (stiffness) were determined, based on the force required to bend the material by 5mm. This was within the linear elastic defamation region and takes the effect of material thickness into account. The data (Table 3) shows that the moulded material gave statistically lower values for this modulus compared to the EPS, indicating that on a unit by unit comparison the pulp moulded material had a lower stiffness the EPS.

In terms of the performance of the products the maximum loads in both the tensile and flexural directions were compared, (Table 4). The data shows that the mean maximum load achieved by the 60/40 mix was significantly higher than that of the EPS and the 80/20 mix. The mean maximum loads for the 80/20 mix and the EPS were not statistically different. This data is based on products with different thicknesses, but it does indicate that the pulp moulded product as produced can match the tensile performance of the EPS, with an adjustment in thickness. The maximum flexural load required to reach 5mm deflection for the 60/40 mix product was statistically significantly (at p=0.05) lower than both the 80/20 mix product and the EPS product. However, the 80/20 mix product with its increased thickness sustained a maximum flexural load that was not significantly different to that of the EPS product.

The combined data from the tensile and flexural tests indicates that products moulded from the straw/paper material in the correct composition can match the performance of the EPS products, although changes in thickness may be required.

3.3.2 Water absorbance

Water absorbancy can be a key atribute in packaging as strength and cohesion of materials can be impaired by too much water. Also, if the material were to be used in food packaging water sorption from the food product would need to be limited. Therefore, the wwater absorbance of the materials was assessed by determining the Cobb value of the materials. A low value shows low absorbance of water, with business paper having a Cobb value of 22-26, unsized paper generally having a Cobb value of 50+ and corrugated cardboard having a Cobb value of 120 - 140 [Jacobs et al 2002]). The data (Figure 4) shows that the EPS had a significantly lower level of water absorption than the moulded products. The moulding process did include the addition of a water repellent, as without it, the water absorbency would be significantly higher. Samples without the additive gave Cobb values of 2664.78 ± 484.2 for the 60/40 mix and 3058.2 ± 113.5 for the 80/20 mix: data that show the effectiveness of the additive. Even though the EPS showed significantly lower water absorbance the values obtained for the moulded materials are on the lower end of the scale for equivalent materials.

Figure 4. Water absorbance of pulp moulded material compared to EPS

3.3.3 Biodegradability

Following the four weeks exposure in the soil test there were clear differences in appearance between the EPS and moulded material, with the moulded material showing clear signs of microbiological growth and biodegradation. On removing the samples from the soil all of the

320 moulded material samples broke into two or more pieces whilst the EPS remained completely
321 intact (Figure 5).
322
323 Figure 5. Comparison of pre soil exposure (A) and post soil exposure (B) of EPS (left) and
324 straw based material (right)
325
326 Mass loss of the samples during the four week exposure was determined and the data (Table
327 5) shows a significant (at p=0.05) difference between the straw based material and the EPS.
328 The data shows that over 20% of the 80% straw based material was degraded after only four
329 weeks soil exposure, implying that the straw based material would be readily compostable.
330 The EPS samples exhibited a slight average weight gain which is attributed to unobserved
331 adhering soil.
332
333

334 4.0 Conclusions

335 The study demonstrated the successful production of material suitable for thin flat pulp
336 moulded materials containing up to 80% straw. Chemical additives were shown to be effective
337 when added at the "wet end" of the moulding process, for example to provide the required
338 water resistance. In terms of intrinsic material properties the moulded material had a tensile
339 modulus higher than the EPS but a lower modulus in bending. In assessing the performance
340 of the products the noted deficiencies in bending strength could be alleviated by using thicker
341 materials. In terms of physical characteristics, the pulp moulded material was heavier than
342 EPS, which may have economic implications. Importantly, it was also shown that the straw
343 based material was biodegradable at ambient temperature in soil, which is a great contrast to
344 the non biodegradability of EPS.

In summary the data presented shows for the first time that it is feasible to produce a flat, thin pulp moulded product derived predominately from cereal straw that possessed the required physical and mechanical performance but that was also biodegradable.

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Table 1. Characterisation of pulp mixes.

Pulp mix	Canadian Standard Freeness (ml)	pH
Base straw	740.6 ± 7.76	5
80/20	725.3 ± 2.8	5.5
60/40	230.0 ± 14.5	8.6

Table 2 Physical characteristics of moulded material compared to EPS.

Sample	Thickness mm	Density kg/m^3
60/40	2.67 ± 0.3	147.15 ± 3.4
80/20	4.54 ± 0.28	120.20 ± 5.5
EPS	3.95 ± 0.1	33.20 ± 0.9

Table 3 Intrinsic mechanical properties of materials

Sample	Modulus in tensile (MPa)	MOE under bending (MPa)
60/40	0.68 ± 0.04	0.028 ± 0.006
80/20	0.47 ± 0.14	0.015 ± 0.005
EPS	0.16 ± 0.009	0.035 ± 0.007

435

436 Table 4. Comparative performance of products

Sample	Max Tensile load (N)	Max Flexural load (N)
60/40	81.99 (11.44)	4.61 ± 0.18
80/20	65.00 (6.48)	2.02 ± 0.25
EPS	67.20 (2.64)	4.02 ± 0.47

437

438

439

440 Table 5. Mass loss of samples after four weeks exposure to unsterile soil

Sample	Mean Mass loss (% based on original dry mass)
EPS	-1.9 ± 1.97
60/40	12.78 ± 3.76
80/20	21.4 ± 3.98

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443 Figure Captions

444

445 Figure 1 Chemical composition of straw pre and post refining labelled straw and fibre
446 respectively (Error bars show standard deviation).

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448 Figure 2. Particle size distribution of pulp mixes

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450 Figure 3. Representative examples of the load/deformation curves for the pulp moulded and
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