# Characterization and Preparation of Wood-Furanic Foams

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#### ABSTRACT:

Fine wood powder/furanic foams were prepared with a strong predominance of the wood component. Low weight and density are important properties of foamed composites. Focusing on preparing light materials, wood foam composites were made using a chemical foaming method and expanded using diethyl ether as the foaming agent. The additives were added to note their effect on the density and mechanical properties, like impact strength and Young's moduli, of the expanded composites. Various tests and scanning electron microscopy analysis were also performed. The foaming agent resulted in closed cells with varied sizes and more or less regular shapes, and with significant density reductions and acceptable loss of mechanical properties.

KEYWORDS: Wood powder, cellular foams, composites, diethyl ether, formaldehyde, blowing agent, bulk density, cell size, mechanical property

## 1 INTRODUCTION

Recent work on resins derived from natural products has led to the development of rigid foams based on natural materials such as vegetable tannins and wood and bark-derived materials coreacted with furfuryl alcohol [1, 2]. While tannins are well known for their various industrial uses [3], foamed composites in which wood powder is the dominant component have excellent performance and characteristics and can find many applications due to economic and environmental reasons. Among these, the availability of low-cost raw materials, their biodegradability and recyclability must be cited. PVC/Wood foams have fond applications in cushioning and packaging industries. Carbon foams are widely used in many industrial and aerospace applications as high temperature thermal insulators and as structural support materials. Hence, these materials have strength and high stiffness to prevent structural collapse during a crash. Foamed composites have better surface definition and sharper contours and corners than their non-foamed counterparts, which are created by the internal pressure of foaming. Foams made out of polymers are advantageous over the neat polymers in terms of materials cost and mechanical properties such as stiffness and strength. Foamed wood composites accept screws and nails like wood, more easily than their non-foamed counterparts [4–9]. Foams are very light solids and, if combined with stiff skins to make sandwich panels, they give structures which are exceptionally stiff and light [10]. The engineering potential of foams is considerable, and, at present, incompletely realized. In very simple terms, foams are composites of solid and a gas. Foams can be prepared by various methods. In the case of rigid foams, one of the points of continuous research in order to upgrade them is the use of foaming agents to maintain strength while maintaining the foam density as low as possible [11–19].

The present work studied the preparation of wood/ furanic foams, which are really composites in which fine wood powder is in the great majority, using a chemical self-blowing approach, and the effect of the proportion of foaming agent (diethyl ether) in their preparation.

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## 2 EXPERIMENTAL WORK

# 2.1 Preparation of Foam Composites

The foaming process consisted of three phases: mixing, expansion and curing. In the mixing process, appropriate amounts of wood fine powder (100 µm), furfuryl alcohol, water, formaldehyde and diethyl ether were mixed together in a beaker and the mixture was stirred mechanically using a Heidolph RZR 2020 stirring machine at 150 rpm for 40 seconds at room temperature. A catalyst composed of 65% solution of para-toluenesulphonic acid (pTSA) was then added to the entire mixture under mechanical stirring. This mixture was stirred until the foam started to expand. After 10 minutes, the skin of the foam was cut out and the remaining structure was taken out of the beaker. The foams so prepared were kept for 2 days before being tested for mechanical properties. This was done to allow solvent evaporation and eliminate the residual blowing agent trapped in the foam. The proportion of the various constituents used for each type of foam is shown in Table 1. The mechanism of foaming is based on the strong exotherm caused by the self-condensation of furfuryl alcohol under acid conditions. For the proportions indicated in Table 1 the temperature of the mixture as a consequence of this reaction reaches a peak of almost 80°C. This marked and rapid increase in temperature brings to boil the diethyl ether that foams the mixture due to its evaporation. Simultaneous to the foaming is the reaction of polymerization of the furfuryl alcohol that leads to foam hardening.

# 2.2 Bulk Density

The samples were cut in  $30 \times 30 \times 15$  mm³ specimens. The bulk density is defined as the weight of the specimen divided by its volume. The foam's cell dimensions were also measured. The average cell diameter was calculated using the empirical formula Av.D =  $(\pi/4)$  D, where D is the average of a certain five number of measured values as given in Table 1. The density of the wood foam is decreased as the amount of blowing agent increases. Diethyl ether is used as a blowing agent to produce foams with a density of between 30

to 80 kg/m<sup>3</sup>. Table 2 shows the recorded bulk density of the various prepared foam samples .

# 2.3 Weight Drop Impact Test

The test is comprised of dropping a mass of 222.9 g on the specimen through specified heights by an impact machine. The mild steel impactor (striker) was designed with a spherical-shaped end of 20.45 mm diameter to simulate non-deforming projectiles. The schematic diagram is shown in Figure 1. The sandwich specimen was placed on a flat surface

**Table 2** Recorded bulk densities of prepared foam samples.

|                  | DE 4  | DE 6  | DE 7  |
|------------------|-------|-------|-------|
| Density (kg/ m³) | 69.70 | 52.86 | 48.25 |

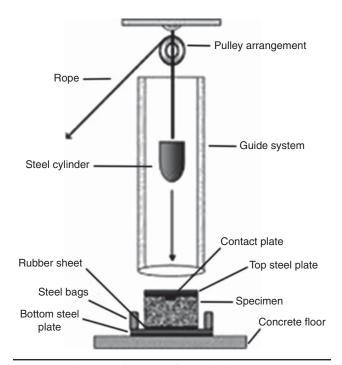


Figure 1 Schematic diagram of weight drop impact test.

Table 1 Amount of various constituents added for various foam specimens preparation.

|      | Furfuryl<br>Alcohol (g) | Water<br>(g) | Formaldehyde<br>(g) | Wood<br>Powder (g) | Diethyl<br>Ether (g) | PTSA (g) |
|------|-------------------------|--------------|---------------------|--------------------|----------------------|----------|
| DE-4 | 13                      | 8            | 10                  | 40                 | 4                    | 15       |
| DE-6 | 13                      | 8            | 10                  | 40                 | 6                    | 15       |
| DE-7 | 13                      | 8            | 10                  | 40                 | 7                    | 15       |



of thick plate to avoid bending, and the striker was dropped from a height of 81.0 mm. The surface of the specimen ( $30 \times 30 \times 15 \text{ mm}^3$ ) developed scattered cracks due to the impact from the striker. The impact energy was obtained from the height dropped. The results of each type of test were measured with the average values of five specimens, as shown in Table 3.

# 2.4 Modulus Test

The samples were cut into  $15 \times 15 \times 30~\text{mm}^3$  specimens. The compressive strength and behavior of the wood foam composite at different amounts of diethyl ether were tested on rectangular specimens. Tests were conducted on a commercially available Hounsfield Tensometer (manufactured by Ensometer Limited, England). The specimen was fixed as a suitable grip attached to the tensometer. The compressive loading of the specimen was then started by rotating the handle slowly at a constant load. The load and displacement values were recorded to obtain the Young's modulus, as mentioned in Table 4.

# 2.5 Scanning Electron Microscopic Analysis

The samples were cut into  $30 \times 30 \times 15$  mm3 specimens. The cells of the wood foam composites at different compositions of diethyl ether were observed by scanning electron microscopy (Phillips PSEM 500) at 200x magnification.

**Table 3** Calculated drop height and impact energies for various foam specimens.

|                         | DE 4  | DE 6  | DE 7  |
|-------------------------|-------|-------|-------|
| Absorption volume (cm³) | 1.36  | 1.36  | 1.36  |
| Dropping height (cm)    | 21.45 | 18.35 | 17.75 |
| Impact energy (J)       | 0.469 | 0.401 | 0.388 |

**Table 4** Various Young's moduli for the prepared foam specimens.

|                          | DE 4  | DE 6  | DE 7 |
|--------------------------|-------|-------|------|
| Young's modulus (MN/ m²) | 2.496 | 1.484 | 1.25 |

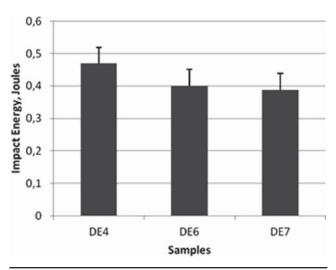
## 3 RESULTS AND DISCUSSION

# 3.1 Weight Drop Impact Energy

When specimen is impacted with weight drop spherical impactor, the impact energy is uniformly distributed along the width of the specimen. The local deformation in the case with the spherical impactor is damaged and compressed only on the front face of the foam and energy was not transferred on the back side of the foam face because foam absorbed the impact energy [2]. Low energy impacts can incur damage, which is hard to detect by visual inspection. However, at certain energy levels, when microcracks are formed on the foam face, the impact damage area is visible. The graph in Figure 2 clearly shows that the impact energy decreases as the amount of blowing agent added is increased. This indicates that the cell size increases with the addition of blowing agent. It is assumed that each cell of foams is bonded perfectly and when impacted the total energy is observed by the cell. After being impacted by a striking object, shock waves are generated from the impact load and propagate along the impact direction. Therefore, density of the foam is decreased because of the condensation of each cell.

# 3.2 Young's Modulus

The mechanical property of the foams depends on the amount of blowing agent. Figure 3 clearly indicates that the Young's modulus decreases as the amount of blowing agent increases. This indicates that the cell size of foam increases with an increase of blowing



**Figure 2** Variation of impact energy with diethyl ether in the sample.



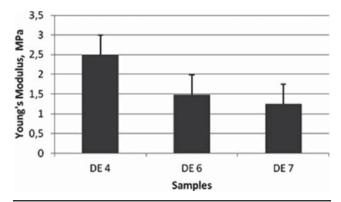


Figure 3 Variation of Young's modulus with diethyl ether in the sample.

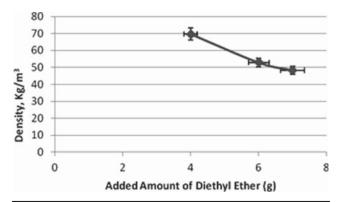


Figure 4 Variation of density with the amount of diethyl ether.

agent, which affects the strength and modulus of the foam composite. The main factor is that the densification rate depends on the density of the foam, which follows the power law [3]:

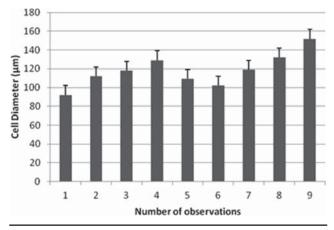
$$\in_{d} = d^x$$

where  $\in_d$  is the densification rate and d is the density.

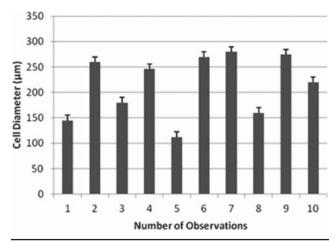
Figure 4 confirms that the density decreases with an increase of diethyl ether blowing agent due to an increase of pore size. The average density value is mentioned in the figure with the percentage variation of 10%. This study confirms the fact that changing the blowing agent changes the modulus properties of foams because densification absorbed the energy under the compression.

### 3.3 Cell Size Observation

Since tannins have a very high reactivity with formaldehyde under acid condition, it is very difficult to find out the correct condition to use tannins to prepare



**Figure 5** Variation of cell diameter with number of observation in DE4 sample.

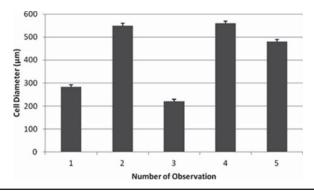


**Figure 6** Variation of cell diameter with number of observation in DE6 sample.

foams. The curing rate of tannin/formaldehyde systems starts at 25°C. The curing reaction should not begin before a sufficiently high temperature is reached to let the solvent play its role of blowing agent. Tanninbased foam requires a significantly higher proportion of furfuryl alcohol to obtain a mixture which is not too viscous and a homogeneous distribution of the tannin. Figures 5–7 show the distribution of cell sizes that was obtained for the different formulations. The results show that the cell size changes from each observation; the average cell size is obtained and indicates that the cell size decreases with the increase of blowing agent, as shown in Figure 8. Scanning electron microscopy investigation confirms that the average cell size increases with increasing amounts of diethyl ether blowing agent.

More details about the microcellular structure of the foams are shown in Figure 9. The closed cells with varied sizes and more or less regular shapes are clearly





**Figure 7** Variation of cell diameter with number of observation in DE7 sample.

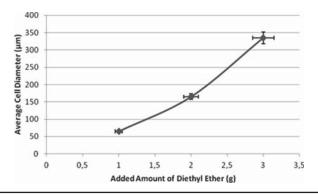
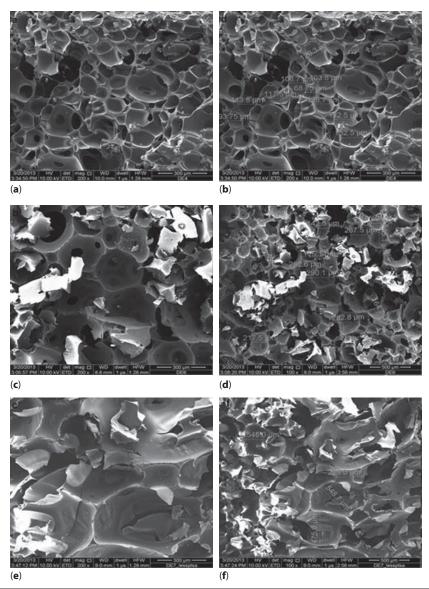


Figure 8 Variation of average cell diameter with diethyl ether



**Figure 9** High magnification scanning electron microscope images of wood foam composites at 200x with different compositions: **(a)** DE 4, **(b)** DE 6 and **(c)** DE 7; and images of wood flour foam composites with different compositions showing dimensions for cell size **(d)** DE 4, **(e)** DE 6 and **(f)** DE 7.

visible in the SEM images at 200x magnification. The cell structures clearly indicate that each cell is made up of edges, cell walls, and windows in the cell walls.

## 4 CONCLUSION

Based on experimental observations, the following results can be concluded:

- i. Diethyl ether added to the mixture is suitable for blowing agent. An increase in the blowing agent content always results in a decrease in the density of the foam. With diethyl ether as the blowing agent, foams of this type with a density from 30–80 kg/m³ can be produced.
- ii. Since the catalyst is pTSA, its impact on mechanical properties and density does not appear to be excessive. The results show that as long as the foams completely expanded, varying the amount of catalyst does not much affect either cell size or foam density (results not shown here).
- iii. The impact energy and Young's modulus are decreased with the increase of blowing agent, which indicates the cell size increases and does not bear the absorption energy and modulus with respect to low blowing agent due to change in density. However, the Young's modulus depends upon bulk density according to the relation:

$$E \propto \rho^a$$

where a is the proportionality constant.

# **REFERENCES**

- 1. X. Li, V.K. Srivastava, A. Pizzi, A. Celzard, and J. Leban, Nanotube Reinforced tannin/furanic rigid foams. *Ind. Crops Prod.* **43**, 636–639 (2013).
- N. Meikleham and A. Pizzi, Acid and alkali-setting tannin-based rigid foams. J. Appl. Polym. Sci. 53, 1547–1556 (1994).
- 3. M. Thebaut, A. Pizzi, and E. Fredon, Synthesis of resins with ozonized sunflower oil and radiata pine tannins. *J. Renew. Mater.* 1, 242–252 (2013).
- 4. A. Pizzi, G. Tondi, H. Pasch, and A. Celzard, MALDITOF Structure determination of complex thermoset networks Polyflavonoid tannin-furanic rigid foams. *J. Appl. Polym. Sci.* **110**, 1451–1456 (2008).

- G. Tondi, A. Pizzi, H. Pasch, A. Celzard, and K. Rode, MALDI-TOF investigation of furanic polymer foams before and after carbonization: Aromatic rearrangement and surviving furanic structure. *Eur. Polym. J.* 44,2938–2943 (2008).
- 6. G. Tondi, A. Pizzi, and R. Olives, Natural tannin-based rigid foams as insulation in wood construction. *Maderas Ciencia Tecnol.* **10**, 219–227 (2008).
- G. Tondi and A. Pizzi, Tannin based rigid foams: Characterisation and modification. *Ind. Crops Prod.* 29, 356–363 (2009).
- 8. G. Tondi, W. Zhao, A. Pizzi, V. Fierro, and A. Celzard, Tannin-based rigid foams: A survey of chemical and physical properties. *Bioresour. Techn.* **100**, 5162–5169 (2009).
- 9. A. Celzard, W. Zhao, A. Pizzi, and V. Fierro, Mechanical properties of tannin-based rigid foams undergoing compression. *Mater. Sci. Eng.* **A527**, 4438–4446 (2010).
- X. Zhou, A. Pizzi, A. Sauget, A. Nicollin, X. Li, A. Celzard, H. Pasch, and K. Rode, Lightweight tannin foam/composites sandwich panels and the cold-set tannin adhesive to assemble them. *Ind. Crops Prod.* 43, 255–260 (2013).
- 11. L.M. Matuana and F. Mengeloglu, Microcellular foaming of impact-modified rigid PVC/wood-flour composites. *J. Vinyl Addit. Technol.* **7**, 67–75 (2001).
- 12. M.C. Basso, S. Giovando, A. Pizzi, M.C. Lagel, and A. Celzard, Alkaline tannin rigid foams. *J. Renew. Mater.* accepted and in press, 1–4 (2014).
- 13. L.M. Matuana, C.B. Park, and J.J. Balatinecz, Cell morphology and property relationships of microcellular foamed PVC/wood-fiber composites. *Polym. Eng. Sci.* **38**, 1862–1872 (1998).
- 14. A.K. Bledzki and O. Faruk, Effects of the chemical foaming agents, injection parameters, and melt flow index on the microstructure and mechanical properties of microcellular injection molded wood–fiber/polypropylene composites. *J. Appl. Polym. Sci.* **97**, 1090–1096 (2005).
- 15. F. Mengeloglu and L.M. Matuana, Foaming of rigid PVC/wood-flour composites through a continuous extrusion process. *J. Vinyl Addit. Technol.* 7, 142–148 (2001).
- 16. G.M. Rizvi, C.B. Park, and L.M. Matuana, Foaming of PS/wood fiber composites using moisture as a blowing agent. *Polym. Eng. Sci.* **40**, 2124–2132 (2000).
- 17. K. Kim and K. Pal, Recent Advances in the Processing of Wood–Plastic Composites, Engineering Materials Nr 32, Springer-Verlag, Berlin. 112–127 (2010).
- 18. X. Li, A. Pizzi, C. Lacoste, V. Fierro, and A. Celzard, Physical properties of tannin/furanic resin foamed by different blowing agents. *Bioresources* **8**, 743–752 (2013).
- 19. M.C. Basso, A. Pizzi, and A. Celzard, Dynamic foaming behaviour of polyurethane vs. tannin/furanic foams. *J. Renew. Mater.* **1**, 273–274 (2013).