

Plant based foam for structural application

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Abstract

Traditionally, polymers and macromolecular components used in foam industry are mostly derived from petroleum. Sustainable society creates demand for use of more renewable feedstocks. Soybean oil is a vegetable oil, composed mainly of triglycerides, is suitable material for foam production. In this study, acrylated epoxidized soybean oil and variable amounts of wood fibres were used in a production of bio-based foam. The developed macroporous polymers were characterized by several techniques, including porosity measurements, nanoindentation testing, scanning electron microscopy and thermogravimetric analysis. It was found that the introduction of wood fibres during the foaming process was necessary to create the three-dimensional polymer foams. Using cellulose fibres has potential as foam stabilizer because it obstructs drainage of liquid from the film region in these gas-oil interfaces while simultaneously acting as reinforcing agent in the polymer foam. The resulting bio-based macroporous polymers possessed a porosity of approximately 56%, and incorporation of pulp fibres did not affect thermal behaviour. Scanning electron micrographs showed randomly oriented pores with irregular shapes and non-uniform pore size throughout the samples.

Abbreviations: AESO: acrylated epoxidized soybean oil; AESO x: acrylated epoxidized soybean oil with x percent of fibres; FE-SEM: field emission scanning electron microscope

Introduction

Polymer foams are a group of lightweight substances, very porous and low density materials. Generally, foams can be classified as flexible or rigid, closed- or open-cell foam (Lee et al. 2011). Microporous polymers are ideal for application where weight saving is critical, such as thermal insulation (Brencis et al. 2011), sandwich structure (Siriruk, Jack Weitsman, and Penumadu 2009), and packaging (Ganjyal et al. 2004). Majority of commercial polymeric foams are oil-based.

Numerous studies are exploring the possibility of developing foams from biomass (Lau et al. 2014; Merle et al. 2016; Schmidt and Laurindo 2010). The great deal of these studies is investigating the possibility of producing polyurethane foams from vegetable oils. Polyurethanes foams are produced by reaction of a polyol with a diisocyanate. Polyurethanes from vegetable oils are based on polyfunctional polyols giving crosslinked products. Properties of such products depend on the crosslinking density of the networks and their chemical composition. Polyols can be synthesized from soybean, palm, castor, sunflower, linseed, and canola oil (Kurańska and Prociak 2016; Mosiewicki et al. 2009; Omrani et al. 2016; Tan et al. 2011). Soybean oil-based polyols can be produced via epoxidation route followed by ring opening, hydroformylation, ozonolysis or transesterification (Petrovic 2008). However, plant based foams still contain a large fraction of fossil oil.

Research attention was given to thermosetting foams, because they were likely to present higher mechanical properties. Vegetable oils can be cured after epoxidation (Altuna, Ruseckaite, and Stefani 2015), acylation (Bonnaillie and Wool 2007), and maleination (Wool and Khot 2001). Bonnaillie and Wool reported preparation of thermosetting foams from acrylated epoxidised soybean oil using pressurized carbon dioxide foaming process (Bonnaillie and Wool 2007). The foams density was controlled by the CO₂ pressure inside the reactor and by the vacuum applied during cure. Resulting foams had mechanical properties comparable with semi-rigid industrial foams.

A mechanical frothing technique for producing macroporous acrylated epoxidised soybean oil foam has been reported by Lee and co-workers (Lee et al. 2011). The authors utilised the mechanical frothing to create gas-liquid foam from soybean-derived renewable monomer mixed with bacterial cellulose nanofibrils dispersed in monomer phase. Mechanically frothed gas-AESO-BC foam was polymerised through microwave heating containing lauroyl peroxide as thermal initiator. The resulting bio-based macroporous polymer had porosity of 60%, and foam stability was higher in case of bacterial nanofibrils presence. This was thought to be to the obstruction of the Plateau border in the presence of bacterial cellulose during capillary drainage of the monomer liquid. Bacterial cellulose contributed to higher mechanical properties as reinforcement. Author Wu reported biofoam composite prepared using short sisal fibres as reinforcement and acrylated epoxidized soybean oil as matrix (Wu et al. 2009). A study of the failure mechanism revealed that adhesion between fibre and matrix was a key issue responsible for damage of the foam, so interfacial interactions were enhanced with surface treatment of sisal fibres. Soil burial tests provided the information that the foams could be biodegradable.

In the present study, we report the preparation of thermosetting acrylated epoxidized soybean oil foams using different fibre content (2, 3 and 4%) as a reinforcement component. Sodium bicarbonate was selected as a low cost, nontoxic, and safe thermally latent foaming agent, and a sodium lauryl sulfate was chosen as a surfactant to stabilize the foam. Usage of volatile toxic styrene monomer and accelerant N,N-dimethylaniline was avoided. The physical and mechanical properties were assessed, and the effect of the reinforcing agent content on foams was discussed.

Materials and Methods

Materials

Enoalfa dissolving pulp, produced by Stora Enso Enocell pulp mill in Finland were used in foam preparation. Acrylated epoxidized soybean oil (>99%, Sigma-Aldrich), *t*-butyl peroxybenzoate (>99%, Sigma-Aldrich), Sodium hydrogen carbonate (Merck), and Sodium dodecyl sulfate (Sigma-Aldrich) were used as received without further purification.

Biofoam preparation

Enoalfa dissolving pulp was blended in PolyMIX machine for 30s, prior foam formation. Polymer foams were prepared by polymerizing the gas-fibre-AESO mixture. Acrylated epoxidized soybean oil was mixed with 1.0% *t*-butyl peroxybenzoate, 4.0% anionic surfactant (CH₃(CH₂)₁₁SO₄Na) and 5.0% blowing agent (NaHCO₃). The wood fibres were introduced into the mixture in different weight fractions (2.0%, 3.0% and 4.0%). Addition of wood fibres increased viscosity of mixture. Chemical blowing agent reacted when 0.5 ml of distilled water was added into the system. Mixture

was placed in an oven for 4h at 80 °C. After polymerization, foams are left at ambient temperature to cool down for 24 h. Post curing procedure was performed at 110 °C for 30 min.

Porosity determination using Argon pycnometer

Grain volumes of the samples were measured using an Ar-gas pycnometer whose operation is based on applying the equation of state of ideal gas. The measurement set up consists of two chambers (sample and reference), pressure gauges and temperature gauges, an Ar-gas supply and a vacuum pump. A more detailed description of the method and equipment is given by Voutilainen et al. 2012. The bulk volumes of the samples were determined by measuring dimension of them using a vernier caliper. The uncertainties of the measurements were determined using general law for propagation of uncertainty. Porosity (ϵ) can be defined using equation

$$\epsilon = \frac{V_B - V_G}{V_B} \cdot 100\%$$

where V_B is the bulk volume and V_G the grain volume of the sample.

The grain-volume measurements results were averages over 10-12 independent measurements. The height of the sample was measured from several (4-6) locations and their average was used when determining the bulk volumes. The given value for pristine soy bean oil sample is highly uncertain due to the isolated pores that were not taken into account using pycnometry. When using Ar-gas pycnometer the gas can only penetrate into pores that are connected to the surface (directly or via other pores) and thus the given value overestimates the true grain volume and underestimates the porosity.

Field Emission Scanning Electron Microscope (FE-SEM)

The morphology of bio-based foams was examined by Leo Gemini 1530 field emission scanning electron microscope with an In-Lens detector. Samples were coated with carbon in a Temcarb TB500 sputter coater (Emscope Laboratories, Ashford, UK). Optimum accelerating voltage was 2.70 kV.

3D Optical Microscope

Non-invasive 3D surface measurements of bio-based foams were examined by Bruker's 3D optical microscope system (Bruker Nano GmbH, Berlin, Germany). Bruker Vision64 software provided functional and streamlined graphical user interface, and comprehensive data collection and analysis.

Nanoindentation analysis

Mechanical properties of biofoam specimens were measured with UBI1 Nanomechanical Test Instrument (HYSITRON, Inc.) using a continuous stiffness measurement in a force controlled mode with a Berkovich type triangular diamond pyramid. The continuous stiffness measurement technique gives access to contact stiffness, hardness, elastic modulus, and creep resistance. Nanoindentation elastic modulus (E) and hardness (H) are defined from the following equations:

$$E = \frac{dP}{dh} \frac{1}{2} \sqrt{\frac{\pi}{A}}$$

$$H = \frac{P_{max}}{A}$$

with P_{max} is the applied load at the maximum depth of penetration, A is the contact area and $\frac{dP}{dh}$ is the slope of the initial portion of the unload curve in the load-displacement plot. At least five indentations were performed on each sample, with a peak load force of 200 μ N and the average

values were calculated. The nanoindentator was calibrated against polycarbonate standard with a maximum standard deviation of 10%.

Thermal analysis

The thermogravimetric analyses of the macroporous polymer composites were carried out in a TGA–DTA thermoanalyser (Q Series instrument). The specimens of 80-100 mg each were heated in corundum crucibles up to 500 °C at a heating rate of 10 °C per min in argon atmosphere.

Results and Discussion

Structure and porosity of bio-based foams

Each foam pore is a volume of gas enclosed in polymer walls. In solid foams, a cell with all its surrounding walls intact is called a closed-cell. When at least two walls are broken during the solidification phase of the foam, the cell is called an open-cell. Polymeric foams consist of a mixture of open- and closed-cells in varied proportions (Bonnaillie 2007). Mechanical and physical properties of thermosetting foams are related to the foam structure, which is controlled by the rates of bubble nucleation, bubble growth, foam aging and polymerization according to the laws of kinetics, thermodynamics and transport phenomena. In this work, we decided to fix the monomer AESO, the type of foaming process and the blowing agent and to focus on reinforcement phase, and effect of fibres on overall porous polymer properties. The pores in the foam were formed by means of chemical blowing agent followed by thermal polymerisation of the liquid monomer. Acrylated epoxidized soybean oil is fascinating due to the high reactivity of the acrylic groups pertaining to easy polymerization via free radicals reaction.

SEM images showing the internal structure and morphology of the pristine AESO foams and reinforced AESO foams are shown in Fig. 1. It can be seen from the SEM images that the pores are randomly oriented with pore shapes that are semi-spherical and mostly highly irregular, from the side view of the materials. Nevertheless, the pore size is highly non-uniform throughout the samples.

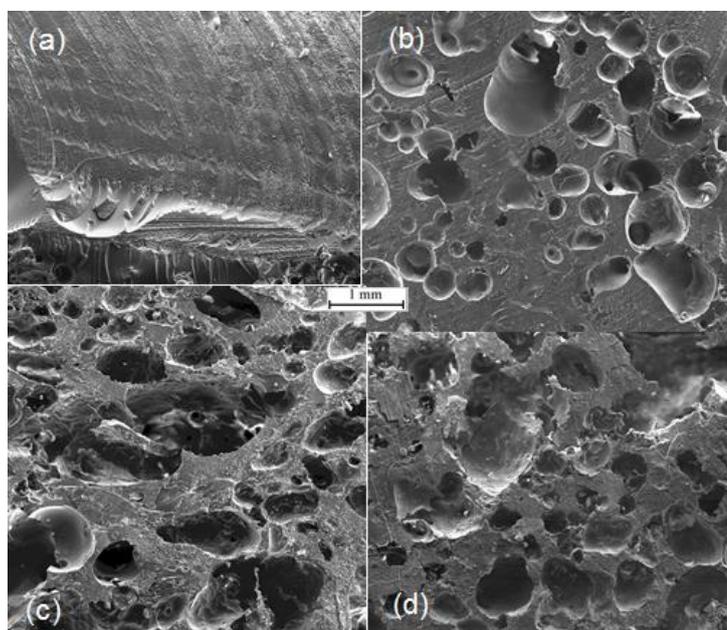


Fig. 1 The SEM images of pristine AESO (a) and AESO reinforced with 2.0% fibres (b), 3.0% fibres (c), and 4.0% fibres (d)

Foam is the dispersion of a gas in a liquid, which creates a characteristic structure when the matrix solidifies. Foams are usually prepared with liquid matrices. Because of the large density difference between air and the liquid, this liquid have tendency to drain. The stability of liquid foams is

governed by the dynamics of the thin interfacial films formed between air bubbles approaching each other. Two main reasons to foam destabilization are gravity and/or capillary drainage which induce film thinning and possible film rupturing unless prevented by repulsive electrostatic or steric forces between the film surfaces (Monnereau, Vignes-Adler, and Kronberg 1999). The foam which did not contained fibres, exhibited rapid destabilisation due to the expected fast kinetics of gravitational drainage, followed by capillary drainage, which ultimately resulted in the full phase separation of the foam. When the monomer phase contained fibres, the kinetics of destabilisation were significantly reduced (Fig. 2). It has been proposed that fibres in the liquid phase will aggregate in the Plateau border, obstructing the flow of the liquid from the foam film (Lee et al. 2011).

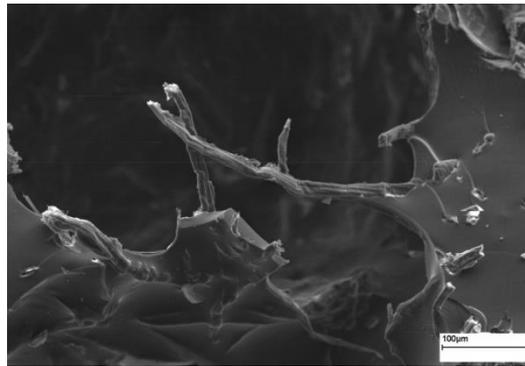


Fig. 2 The SEM images of wood fibres in AESO 4 foam

It can be seen on Figure 1 b, c and d that the addition of cellulose fibres resulted in increase of cell size likely due to the increased number of nucleating sites induced by the fibre surfaces (Bergeret and Benezet 2011). At the same time, the fibre reinforced foams have less uniform pore structure, which should be related to the fibre distribution within the polymer matrix and the fibre-matrix interactions. It is suggested that as a result of local fibre-matrix debonding holes are induced, where the gas loss hinders the cell growing ability, and thus non-uniform distribution of cell size is obtained (Fig. 3). With increasing cellulose fibres loading, the monomer phase became more viscous and the expansion of the gas bubbles in the monomer phase was hindered. This state of the bubbles results in the randomly orientated pores.

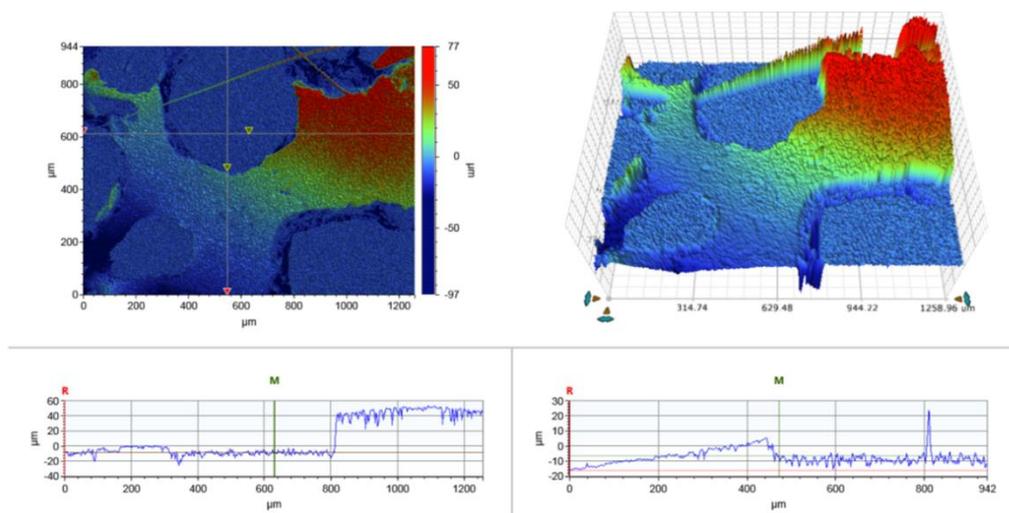


Fig. 3 The 3D images of AESO foam reinforced with 2% fibres

Porosity is one of the important parameters that govern the physical properties of polymer foams. Grain volume, bulk volume, grain density and bulk density along with the calculated porosity of bio-based foams are tabulated in Table 1. It can be seen that the porosity increased from 4.5% to 57.0% with incorporating cellulose fibres. The introduction of wood fibres into the monomer phase resulted in stabilizing the gas-soybean oil interface during thermal polymerization, resulting in rise of total porosity. The highly porous nature of the bio-based foam AESO 3 is a direct result of the mixing process to introduce air bubbles into the bio-based soybean oil resin. It can also be

seen from Fig. 1 c that porous polymer had the largest cell size compared to AESO 2 and AESO 3 foams. The larger pore size observed in AESO 3 is due to the phase separation of the liquid bio-based foam, as liquid foams are inherently unstable (Lau et al. 2014).

Table 1 The porosity, volumes and density of the macroporous polymers

	Porosity ϵ [%]	Grain volume V_G , [cm ³]	Bulk volume V_B , [cm ³]	Grain density [g/cm ³]	Bulk density [g/cm ³]
AESO	4.5±4.4	1.58±0.05	1.65±0.06	1.55±0.04	1.48±0.04
AESO 2	57.0±1.8	1.92±0.05	4.45±0.15	1.28±0.02	0.538±0.004
AESO 3	58.3±1.5	2.16±0.05	5.17±0.15	1.134±0.013	0.474±0.003
AESO 4	54.2±1.8	2.08±0.05	4.55±0.15	1.177±0.014	0.538±0.004

The density of solid polymeric foams typically range from 0.016 g/cm³ to 0.960 g/cm³, according to the requirements of a broad range of applications. High-density foams usually have a high mechanical strength and are used as lightweight structural components for furniture, construction and transportation. Medium-density foams are mostly used in packaging industry, but with satisfactory rigidity they can be utilized in automotive industry. Foams produced in this study are on the border between medium and high density materials. Furthermore, for structural applications, a strong resistance to deformation is desired, and medium to high density foams are preferred (Bonnaillie 2007).

Mechanical properties of bio-based foams

Mechanical property of the foams based on AESO and AESO reinforced with cellulose fibres was studied by nanoindentation method. Since foam cell wall material is not suitable for conventional compressive and tensile strength testing nanoindentation was chosen as a method of nanomechanical characterization. It can provide useful information of the cell wall properties like nanoindentation hardness and reduced modulus. The values of the nanoindentation hardness and the reduced modulus of the cured foams were determined from indentation curves and are shown in Fig. 4. It can be found that the indentation hardness of the foams based on AESO was higher compared to that of AESO reinforced with cellulose fibres, the same trend is observed for the reduced modulus.

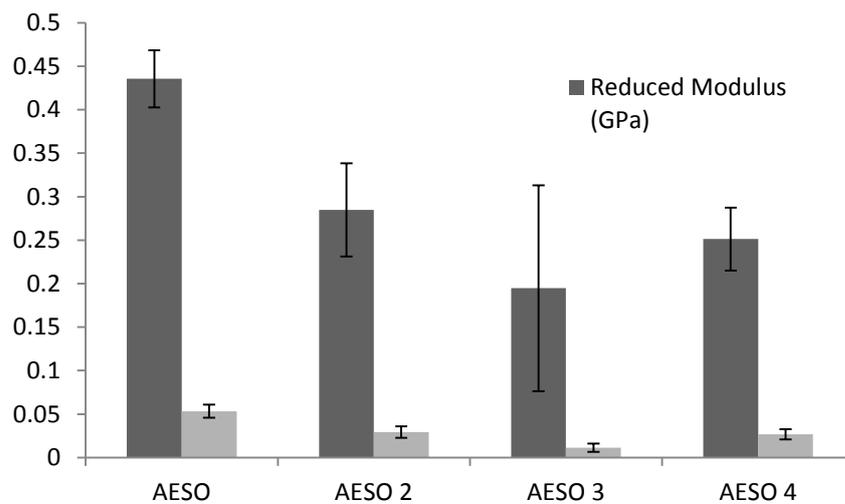


Fig. 4 The indentation hardness and reduced modulus for pristine AESO and AESO reinforced with cellulose fibres foams

It is known from the literature that polymer AESO will have higher local hardness values than the AESO foam (Chen and Liu 2013). This increase in hardness value can be explained due to indenting compact AESO material. Regarding the load-displacement curves for the pristine AESO

foams, all of them presented similar penetration depths and slopes, indicating that the AESO foam maintained its properties from the inner to the outer regions.

Porous materials show several types of heterogeneity at microscale. The heterogeneity can originate from unreacted components or from chemical reactions that are evolving after the mixing. AESO foam is a material with both these heterogeneities and material properties of the foam cell walls are affected by adding stabilizing and foaming agents to mixture during the manufacturing process. The overall behaviour of AESO foam is directly dependent on the formation of the individual phases and their micro-properties. With decreasing pore sizes and pore distances, the influence of the interphase material around the pore becomes more important and characteristic changes in the nano- and micro-deformation mechanisms appear (Michler and von Schmeling 2013). Under load, structural openings, variations of local composition or orientation, nano sized pores degrade the mechanical properties of material.

The effect of the fibre content on mechanical properties can be described based on experimentally determined results. Increasing fibre content for 2.0% to 3.0% induces decrease of the hardness and reduced modulus. Contrarily, increasing fibre content for 3.0% to 4.0% in the matrix showed increase in hardness and reduced modulus. However, the specific data were dependent on specimen surface. The degradation of mechanical properties for AESO 3 can be linked to highest porosity and pore size these samples have compared to AESO 2 and AESO 4. The investigations indicate that the mechanical properties can be enhanced at higher fibre content. However, the hardness value and reduced module of AESO reinforced fibre foams are similar to values for pure polyurethane foam used in structural applications such as the automobile and wind mill blade industries (Gupta et al. 2015).

Thermal behaviour of bio-based foams

The thermal degradation behaviour of the macroporous polymer is shown in Fig. 5. It can be seen that all the samples undergo single step degradation in argon atmosphere. Random polymer chain cleavage occurred during the degradation of acrylated epoxidized soybean oil (Behera and Banthia 2008). The incorporation of cellulose wood fibres into the specimen did not alter the degradation behaviour of final bio-based foam. This might be due to the low cellulose fibre content in the sample. The onset degradation temperature determined from TGA analysis of the foams was found to be 380 °C for all samples. The residual carbon content was found to be slightly higher with samples containing cellulose fibres compared to pure soybean oil polymer. The increase in the residual carbon content can be explained by the carbonisation of cellulose fibres in the mixture. TGA thermogram clearly indicates good thermal stability of the cured foams up to 300 °C with a minimum amount of weight loss, which may be due to the minimum amount of unreacted components.

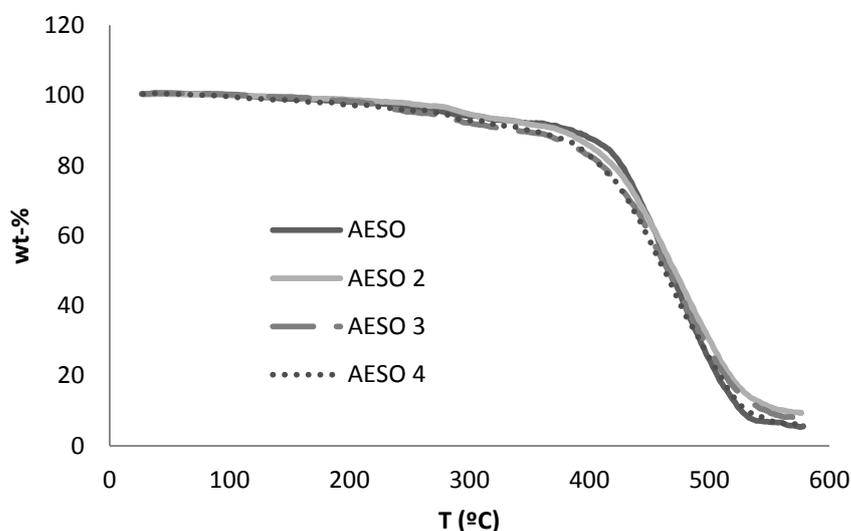


Fig. 5 The thermal behaviour of pristine AESO and reinforced AESO bio-based foams

Conclusions

Bio-based foams from natural fibres and an acrylated epoxidized resin were produced in this study. The resin was mixed with different concentrations of cellulose fibre, additives and thermally polymerized. The results show that it is possible to produce bio-based foams with satisfied mechanical properties without adding to the resin a reactive comonomer like styrene. The resulting bio-based porous polymers possessed a porosity of approximately 56% and were in a range of medium-density foams. It was found that the stability of the gas-AESO interface was poor in comparison to the gas-AESO interface containing cellulose fibres. Both hardness and reduced modulus show that with increasing the fibre content up to certain amount desired mechanical properties can be observed. The incorporation of cellulose fibres into the polymeric foams exhibited lower values of the hardness and reduced modulus compared to pristine AESO foam due to difference in porosity and pore size. However, a further increase of pulp fibres content to 4 wt% resulted in an increase of the hardness and reduced modulus in comparison to 3 wt% fibre content in foam. This was attributed to the reinforcing effect of the wood fibres. Thermal behaviour of macroporous polymer was not affected by the addition of wood fibres. SEM images showed randomly oriented pores with irregular shapes and non-uniform pore size throughout the samples.

Acknowledgments

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