THE EFFECT OF COCONUT FIBERS ADDITIVE AS NUCLEATING AGENT ON MICROCELLULER FOAM PLASTIC STRUCTURE PROCESSED WITH SUPERCRITICAL NITROGEN

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ABSTRACT

Foam plastic with cell size which is smaller then conventional one has been processed by the using of supercritical fluid as blowing agent. Coconut fibers were used as additive and nucleating agent. The investigation was performed under constant saturation temperature at 443.15 K for various saturation pressures from 10 MPa up to 22 MPa. Quick heating methode was applied in foaming process to ensure the foaming near the saturation concentration of gas, and at the higest supersaturation condition for cell nucleation. The results show that, bulk density of foam decrease with an increase of saturation pressure, for both polypropylene pluff and PF1000. Variation of saturation pressure has lead to change of cell diameter and cell number density of the resulting foam product. Volume expansion ratio of foam produced from polypropylene pluff is 50 % higher than polypropylene PF1000

Keyword : polypropylene, foam, coconut fibres, microcelluler plastic, supercritical

INTRODUCTION

Foam plastic is a material at which numerous bubbles have been created in the bulk of the plastic. Microcelluler plastic is a foam material that has several superior mechanical and thermal properties over conventional foam plastic. The usual characterics of this material are cell sizes in the range of 0.1 to 10 micrometers, cell densities in the range of 10^9 to 10^{15} cells/cm^3, and specific density reduction between 5% to 98 % . A well known method for generating of this materials is performed by saturating a polymer with a safe blowing agent (CO_2 or N_2) under a saturation temperature and a saturation pressure. The saturated polymer-gas solution is then brought into a thermodymanically unstable state, where the solubility of the gas is instantaneously reduced by decompression and/or the heating process [1]. Since the application of HCFCs, HFCs, and other volatile organic compound is to be prohibited in the future, the use of nontoxic blowing agent in microceluller plastic processing is a paper choice because of the necessity of applying a safe process and producing environmentally safe products.

The development of microceluller plastic production is addressed to obtain foam material with high cell density, small cell diameter, and high volume expansion ratio. Generally, there are three main processing step : gas polymer salution formation, subjecting the solution to thermodynamically unstable state to promote nucleation of cell, and controlling the cell growth to obtain the desired cell structure. Experimental results showed that the cell morphology could be changed by the variation of process condition [2].

Colton and Suh presented theoritical and experimental studies on the polystyrene-nitrogen system in the presence of additives in a polymer. They extended the classical nucleation theory by considering free volume effect due to additives and gases in a solution. They found that there were three regions of nucleation, i.e., a homogenous region below the solubility limit of te additive of the zinc stearate in PS (approximately 0.3%), a heterogenous region above the solubility limit and a mixed mode region around the solubility limit. However, the theoritical treatment showed an overprediction at high pressure and an underprediction at low pressure [3]. Ramesh *et al*., presented the dynamic of cell growth under various process conditions. They reported that raising the heating temperature increased the cell size because of the decreasing melt viscosity and increasing diffusivity of the gas in the polymer [4]. Sumarno *et al*., have reported a new method in generating microceluller plastic called quick heating method. Quick heating is the heating of a polymer gas solution without a time interval after the decompression process by the heat capacity of the saturation vessel [5].

The objective of this study is to investigate the effect of coconut fibres additive as nucleating on the microceluller foam plastic structure, which are processed at the melting temperature of polypropylene and for various saturation pressure. In this work, processing at high temperatures, even near the melting of the polymer, provides two benefits, i.e., the saturation time is shorter than at low temperature, and foaming process can be proceeded in high concentration of dissolved gas. The supersaturation degree might be high and cause high nucleation rate of bubbles inside the plastic.

EXPERIMENTAL

Materials

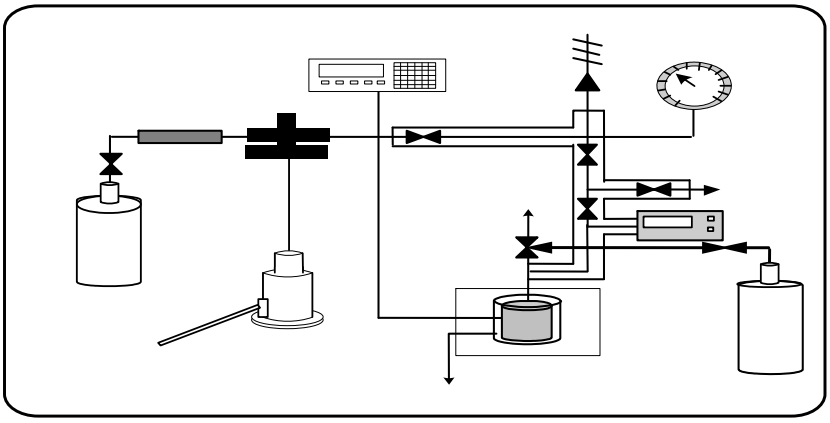
Polypropylene (PT Pertamina Plaju Indonesia, MFI of 10 g/10 minute at 230^0C and spesific gravity of 0.91 g/cm^3) was used with N_2 as a blowing agent (PT Trigases Indonesia) and CO_2 as a cooler gas (PT Trigases Indonesia). The coconut fiber used as an additive were taken from local product of Malang, Indonesia. The samples (polypropylene and an additive) were mixed in internal mixer apparatus (Haake Rheocord 90). The blend were then brought into a sheet with a thickness of about 1.3 mm, which were then cut into 10x10 cm pieces.

Apparatus and Procedure

A schematic diagram of the experimental apparatus used in this study is shown in Fig.1. The samples were placed in a stainless steel saturation vesel immersed in an air bath (EYELA: WFO-450SD). The setup apparatus and polymer sample placement in the saturation vessel is shown in Fig.2. At first, the N_2 gas was delivered into the saturation vessel slowly (from gas booster) up to a desired saturation pressure at a saturation temperature. When the required saturation time was reached, the vessel was immediately decompressed to at atmospheric pressure. The saturation time was determined by calculating the time needs for the center of the sheet sampleto reach over 98% of the saturation concentration. In this work, the saturation time was taken twice of the calculated value, to ensure the saturation condition of nitrogen in polymer. After the decompression, the samples were kept in the saturation vessel for a desired heating heating time to be heated by the heat capacity of the trays and vessel. Flowing of CO_2 gas in to the vessel terminated the heating process and froze the internal foam structure. Expansion of CO_2 from high pressure in to vessel under athmospheric lead to decreasing of vessel inside to be freezed. The samples were kept and freezed farcture in nitrogen liquid. The fractured surface was coated with gold and then characterized using SEM (Scanning Electron Microscope) JEOL model JSM-T330A.

RESULT AND DISCUSSION

The experimental results were characterized to understand the sturctural change of foam for various process variables.



1. N_2 cylinder
2. Filter
3. Gas Booster
4. Digital thermometer
5. Safety valve
6. Pressure Gauge
7. Digital Pressure Indicator
8. Saturation vessel
9. CO_2 cylinder

Fig. 1. Schematic diagram of microceluller plastic experimental apparatus

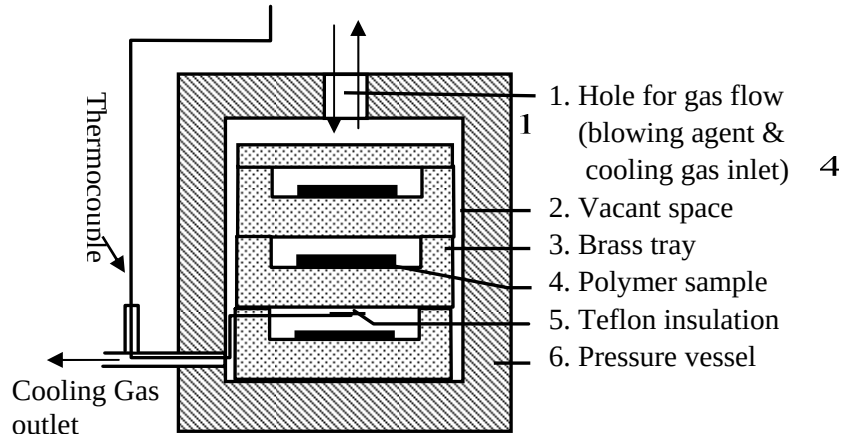
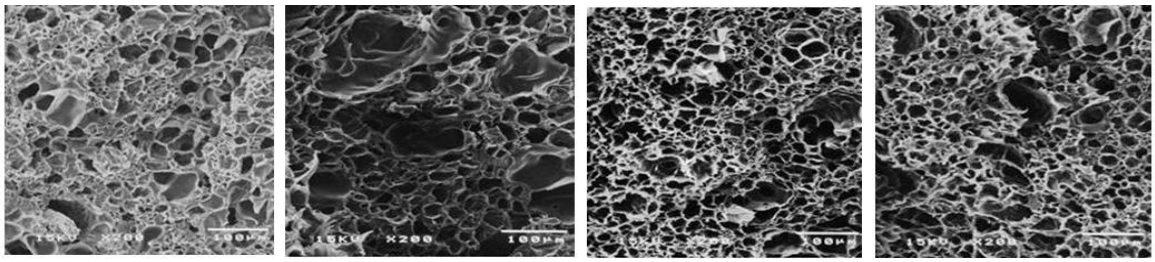


Fig. 2. Set up of polymer sample placement in the saturation vessel under Quick-heating process

Effect of Saturation Pressure

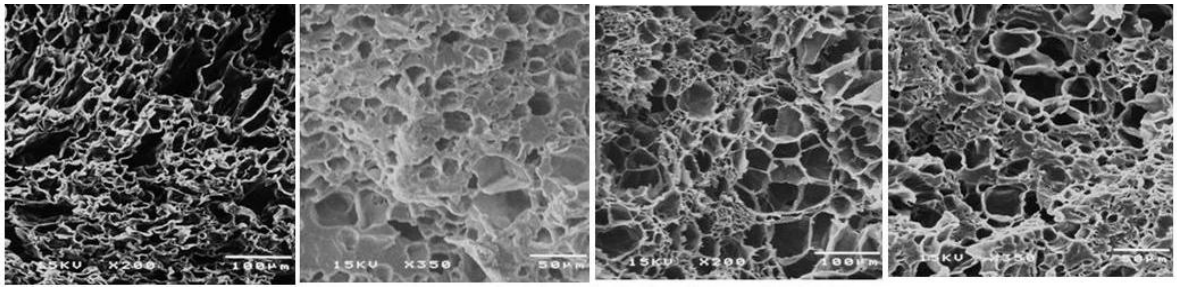
Variation of the microceluller foam structure processed under various saturation pressure and in the presence of coconut fibres additive are presented in potograph of Fig. 3. and Fig. 4. Figure 3 show the foam structure of sample made form polypropilene pluff and 1 % by weigth of coconut fibres. The number of cell and cell size change by saturation pressure. It is seen that the foam structure of sample processed under 18 MPa and 20 MPa are similar. The presence of 1 % by weight of coconut fibres was not affect to the bubble nucleation although the dissolved gas was increased.



(a) 14 MPa (b) 16 MPa (c) 18 MPa (d) 20 MPa

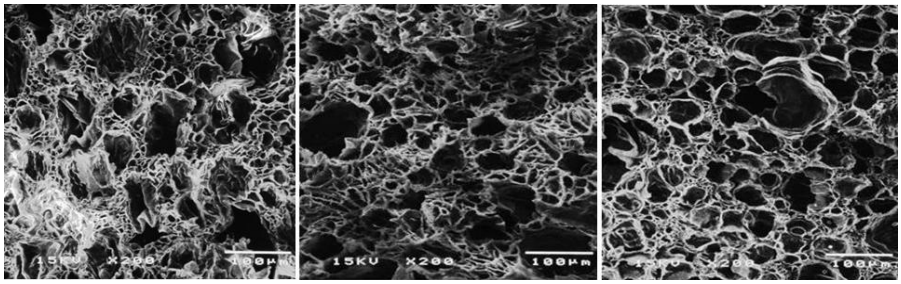
Fig. 3 . SEM photograph show the effect of saturation pressure on change of foam structure, for PP pluff-N2-1%Coconut fibre system.

Figure 5 is photogaph of foam samples processed from polypropilene pluff and coconut fibres of 5 % by weight. Increasing of additive concentration lead to significant change of internal foam structure.



(a) 14 MPa (b) 16 MPa (c) 18 MPa (d) 20 MPa

Fig. 4 . SEM photograph show the effect of saturation pressure on change of foam structure, for PP pluff-N2-5%Coconut fibre system.



(a) 14 MPa (b) 16 MPa (c) 18 MPa

Fig. 5 . SEM photograph show the effect of saturation pressure on change of foam structure, for PP pellet-N2-5%Coconut fibre system.

The number of cell nucleated is higher by increasing the amount of additive, which meant that it affect to the presence of nucletaion site by incresing of the presence of fibres inside the polymer. Comparison of cell structure when processed using polypropilene pellet is shown in Figure 6. Pellet material is mean pocessing further of PP pluff by addition some additive to increase the properties and also it processing behaviour. The photograph show that pellet samples can be foamed easier then the pluff sample. The photos are analyzed using image sofware to get cell size and cell density, as presented in Figure 6 and Fugure 7.

There are two kind of cell, macrocell and microcell presence together. The resulting bubbles could be produced by two possible wasy; nucleation of cell in the area where there are no fiber additive presence, and the growing of the nucleation sites in the positions of fiber particles presence in polymer matrix. The latest one is more priority to be a stable bubbles and grow firstly, and the number is depend on the amount of fiber particle. It is beacuse the presence of uncovered path between particle and surrounded polymer and also the crack inside of the particle caused by interfacial tension between particle and polymer. Those positions will be contained by gas cluster along saturation process. The macrocells occured in the amarphous area and moreover by the presence of particles additive. The microcells could be occured in both amorphous and crystalline area by homogeneous and/or heterogeneus nucleation scheme.

In this study we analyzed the microcell area only and got a cell size and number of cell density based on the photograph of SEM. The bubble size decreases with an increase in saturation pressure, as depicted in Fig 6 and Fig 7. As mention previously above that increasing saturation pressure cause the decreasing of viscoelastic properties of polymer-gas solution. Nucleation rate might be increase, but the existence of competition between nucleation of new cells and grwowing of the pre-existing gas clusters lead to the decreasing of nucleation rate and cause a decresing of cell density. The phenomenon over than 18 MPa was not fully understood yet. Since the bulk density and cell size decrease, and volume expansion ratio constant, it's meant that the sample undergo a shringking processs. The structural change already explained by Sumarno *et all*. [5]. Processing at higher pressure

cause nucleation and growth rate increase that lead to an increase of the number of cell density, but unification and collapse cause deterioration of cell structure as clearly depicted in Figure 5.

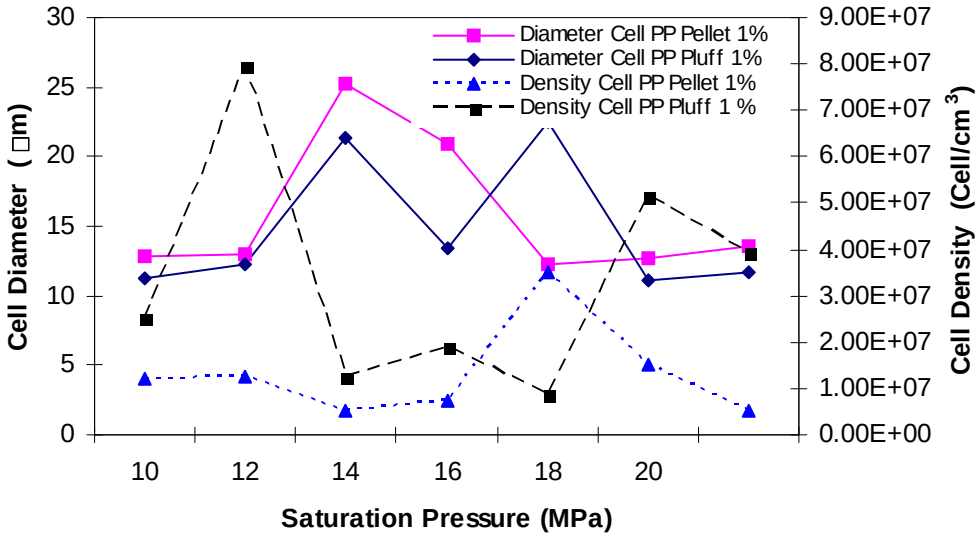


Fig. 6 The effect of saturation pressure on microcell structure for coconut concentration 1%

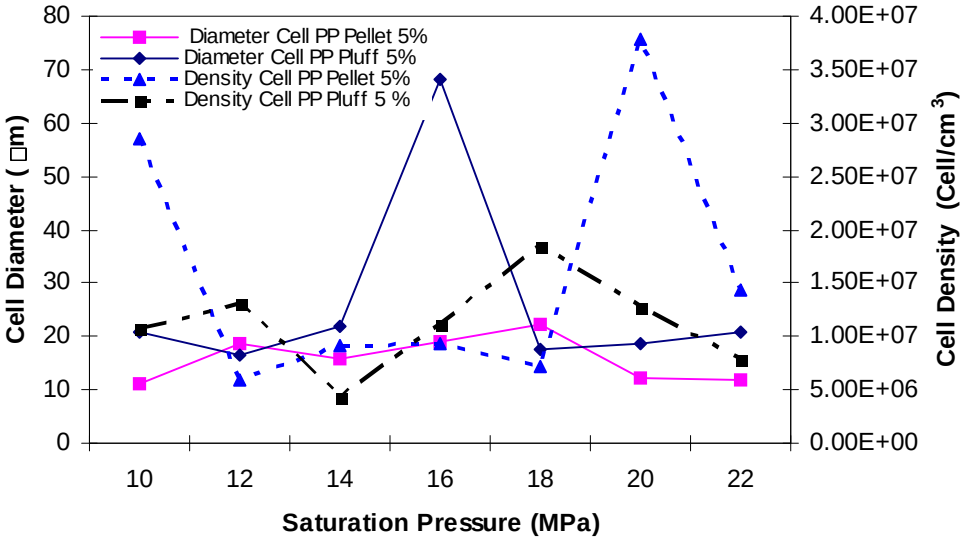


Fig. 7 The effect of saturation pressure on cell structure for coconut concentration 5%

Effect of Saturation Pressure on Volume Expansion Ratio

In order to study the influence of saturation pressure, the polymer was saturated under various pressure for saturation temperature of 170 ^{o}C. The effect of saturation pressure on rasio expansion volume is shown in Fig 8. Volume expansion ratio of foam product is increase with an increase of saturation pressure, with the pluff type is higher than the pellet. Since the solubility of gas increase with saturation pressure, the viscoelastic properties of polymer-gas system decrease. The foaming process could be easily proceeded moreover by the presence of fibres inside the polymer matrix. The absence of additive in the pluff samples cause the bonding between the chains were weak and lead to the lowering of viscoelastic properties of the solution. Nucleation and growth of cell can be proceeded at higher rate then the pellet samples. But, both plastics type have small volume expansion then amorphous polymer as reported by Sumarno et al. [5].

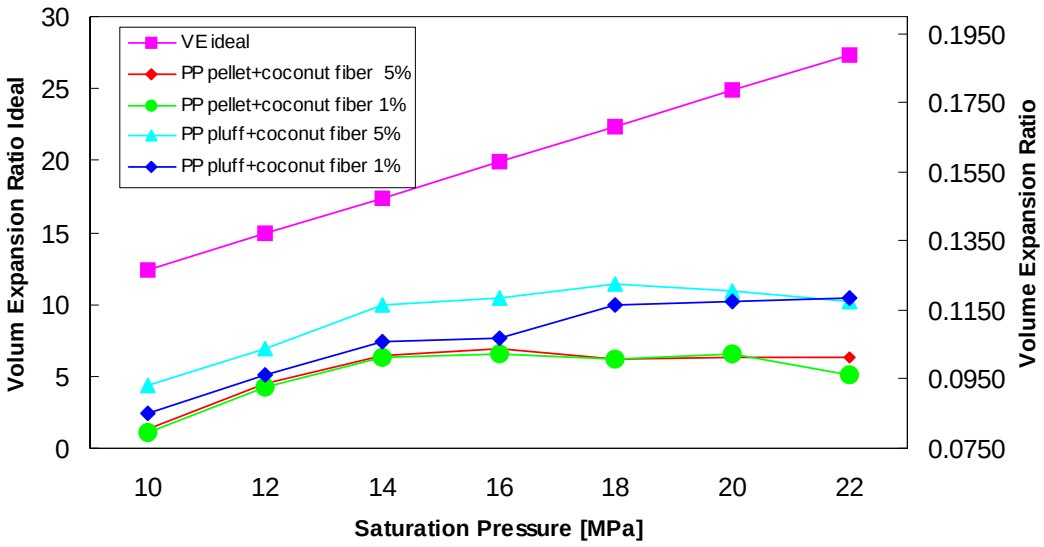


Fig. 4 The effect of saturation pressure on volume expansion ratio

CONCLUSION

Microcellular foam plastic can be produced from polypropylene with coconut fiber as additive. Processing under various saturation pressures, and under saturation temperature of the melting point of polypropylene affect to the change of cell structure. The volume expansion ratio increase with an increase of saturation pressure. The using of coconut fibers particles as filler affect to the cell nucleation and growth significantly.

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