

PREPARATION AND CHARACTERIZATIONS OF PALM OIL BASED RIGID NANOCOMPOSITE POLYURETHANE FOAM

Firuz Zainuddin¹, Sahrim Ahmad¹, Rozaidi Rasid¹ and Syed Nuzul Fadzli Syed Adam²

¹*Materials Science Programme, School of Applied Physics,
Faculty of Science and Technology, Universiti Kebangsaan Malaysia,
43600 Bangi, Selangor.*

²*School of Materials Engineering, Universiti Malaysia Perlis, 02600 Arau, Perlis.*

ABSTRACT

The objective of this research is to study and analyze the basic and mechanical properties of polyurethane (PU) foam. PU foams were initially synthesized with reaction of natural oil polyol and isocyanate with ratio of 1:1.1 by weight. Water was used as the blowing agent and appropriate surfactant and catalyst were added to ensure better performance of final product. Cloisite 30B was added as filler and the percentages were varied from 1 to 5 wt%. The basic properties of polyurethane foams were determined, such as free rise density (FRD), foaming time and rheological index. Clay dispersion in polyurethane nanocomposites was investigated by X-ray diffraction method. Mechanical properties of foams were evaluated using uniaxial compression test where the energy absorption values were determined. Aluminium-polyurethane foam (Al-PU) sandwich composite was prepared and flexural test was carried out to examine the effect of skin attachment to PU foam. The results showed that with increasing nanoclay content resulted an increment in FRD to 74.6 kgm^{-3} , rheological index to 1.07 gcm^{-1} and foaming by almost 133% for the addition of 5wt% nanoclay, respectively. Increasing in percentages of nanoclay filler in PU foam nanocomposite is also found to improve the absorption energy from 89J for pure PU foam to 118J for PU foam with 5wt% nanoclay. It was found that PU foam nanocomposite has better failure deformation as compared to sandwich composite with flexural extension that increase up to 9.4mm. However, flexural stress for sandwich composite are higher up to 3.6MPa. Furthermore, Al sheet acts as ductile skin to PU foam and prevent samples from rupture rapidly or prevent the formation of brittle fracture.

INTRODUCTION

Closed-cell polymer foams are used in packaging, thermal insulation and buoyancy aids. Polyurethane foam, which is one of the most widely used cellular polymers are ubiquitous in our daily lives. Their structure and properties have been extensively explored by many researchers [1,2,]. Industrial PU foam was prepared from reaction of two basic materials, which are an oligomeric polyol from petrochemical-based, and diisocyanate with addition of blowing agent [3]. Nevertheless, with the increasing awareness of petroleum consumption crisis, the replacement of present polyol is highly needed. Recently, polyol from palm oil has been found as replacement for conventional

petrochemical-based polyol [4].

Our aim is to produce polyurethane foam that is an extremely versatile group of polymers. It can be produced in wide range of densities, crosslink densities and stiffnesses, from very soft to very hard structure. Ionescu [3] has divided polyurethanes into two main categories that are elastic polyurethanes, e.g., flexible foams, elastomers, coatings, fibres etc., and rigid polyurethanes, e.g., rigid polyurethanes foams, structural foams, wood substitutes, etc.

One of the ways to improve polymer foams is making it as a sandwich with foam as the core. The sandwich concept is based on two main ideas: increasing the stiffness in bending of a beam or panel and doing so without adding excessive weight [5]. Sandwich panels are often used in applications where weight-saving is critical, eg., in aircraft, portable structures and in sports equipment. Additionally, skins or face materials that is commonly used are aluminium or fibre-reinforced composites [6]. This paper discussed newly developed PU foam nanocomposite from palm oil.

METHODOLOGY

Materials

The commercial clay used in this study is Cloisite® 30B from Southern Clay Products, which is a quaternary ammonium salt modified natural montmorillonite having particle size between 2-13µm. To prepare the polyurethane foam, polyol based palm oil was used along with Maskimate 80, which is an isocyanate. Maskimi Polyol Sdn Bhd. supplied both polyol and isocyanate. Catalyst used in this research is TEGOAMIN PMDETA (N,N,N',N'',N'''-Pentamethyldiethylenetriamine) which is commonly used as catalyst for rigid foam. TEGOSTAB B8407, which is a non-hydrolysable polyether polydimethylsiloxane copolymer was used as surfactant. Degussa/Goldschmidt Chemical Corp. supplies both catalyst and surfactant. To produce sandwich composite, aluminum sheet with 0.5mm thickness were used as skin while a mixture of Epikote 828 and Epikure F205 at 1:2 by weight was used as adhesive to adhere the skin with polyurethane/nanoclay foam.

Synthesis of polyurethane/nanoclay foam

Nanoclay was dehydrated in an oven at 80°C for 24 hours. Polyurethane/nanoclay foams were prepared by mixing clay with one monomer, which is polyol by a high shear mixer at 5000 rpm speed. The percentages of clay were varied from 1 to 5 percent by weight. Catalyst and surfactant were added with polyol after it was first mixed with clay. Excess isocyanate was used to assure the complete reaction of polyol.

Preparation of sandwich Al-PU foam

The composite skins were bonded to the polyurethane foam cores using mixture of resin and hardener. Here, the adhesive was applied to both the skin and core materials and the resulting structure was placed under heavy weight for overnight.

Test methods

The flow properties of the foam were evaluated via rheological test and free rising test. The foams were deformed using Instron Series testing machine at the crosshead speed of 10mm/minute in compression test. Flexural test was done using Testometric universal testing machine at the crosshead speed of 5mm/minute. Each test was done in quintuplicate.

RESULTS AND DISCUSSIONS

PU Nanocomposites

Foaming is one of the crucial parts in PU foam's processing. The rapidity of foaming process will influence condensed properties of the foam in a mould. Table 1 shows the foaming time properties of nanocomposite foam at various stages. It shows that with increasing of nanoclay content, resulted in slightly increase of cream and fiber time but significantly increased in tack free and rise time. This is also the effect of nanoclay dispersion during the foaming process. Generally, nanoclay is very light material and well distributed in the mixture of starting materials. However, because of its nano size, even the minimum percentages can weigh up the viscosity of mixture, thus the slower the reaction between isocyanate and water which will decelerate the production of carbon dioxide gas.

Table 1: Foaming time of nanocomposite PU foams.

Nanoclay %	Cream time, s	Fiber time, s	Tack free time, s	Rise time, s
0	25	44	66	111
1	27	46	74	132
2	28	57	87	138
3	30	58	115	148
4	34	74	142	172
5	36	78	154	210

For high performance materials, bulk density is more important and crucial. In this study, the addition of nanoclay in polyurethane foam has increased the bulk density of nanocomposite foam. This is probably due to exfoliation of nanoclay into cell wall of polyurethane foam, which causes the foam to be more denser. As depicted in Figure 1, addition of nanoclay has increased the density from 0.132g/cm³ for control polyurethane foam to 0.185g/cm³ with an addition of 5% nanoclay into polyurethane foam, which resulted about 40% enhancement of density.

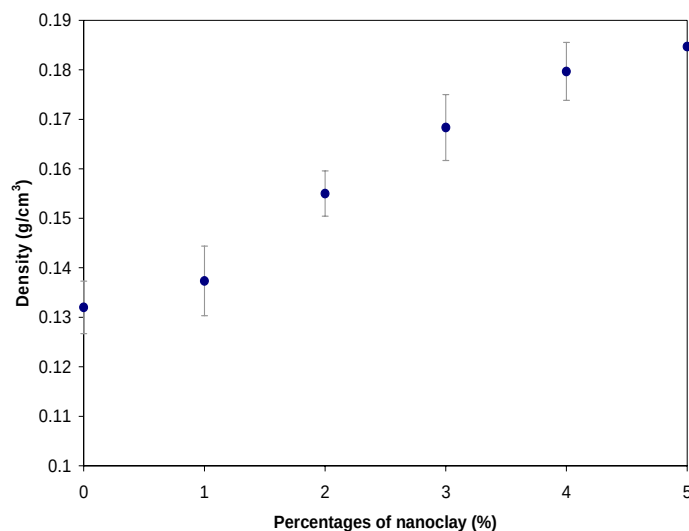
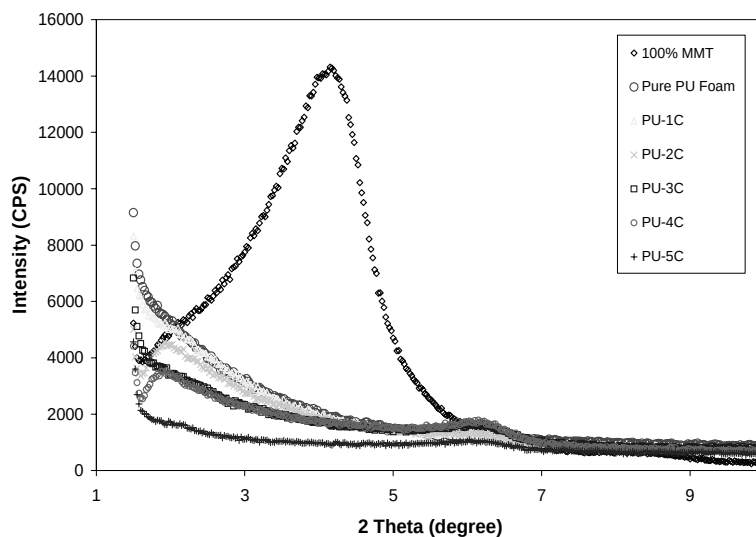


Figure1 : Effect on additional of nanoclay to PU foam density

XRD diffractogram of the raw clay and polyurethane/nanoclay foam are shown in Figure 2. There are no clear peaks found in nanoclay/PU foams, which confirmed good clay dispersion. However, PU-4 shows a clear peak at $2\theta = 6.2^\circ$ which indicates an agglomeration of clay in certain parts in the foam. Although the nanocomposite with 4% of nanoclay is not the highest filler percentages, it is believed that the clay orientation and dispersion is slightly affected by the foaming process.



(b)

Figure 2: XRD diffractogram

Rheology index of the foams increases up to 45% for nanocomposite with addition of 5% nanoclay compared to unfilled PU foam as shown in Figure 3. It shows that with

increasing percentages of nanoclay, the fluidity property during foaming process is improved. The dotted curve in Figure 3 shows the free rise density (FRD) of foams. It obviously shows that PU foam without clay has higher FRD than nanocomposite with 1% and 2% nanoclay, suggesting that the dispersed clay particles act as heterogeneous nucleation sites during cell formation [1]. In contrast, FRD of 3% to 5% clay PU increase rapidly due to the increase in viscosity and weight of foam.

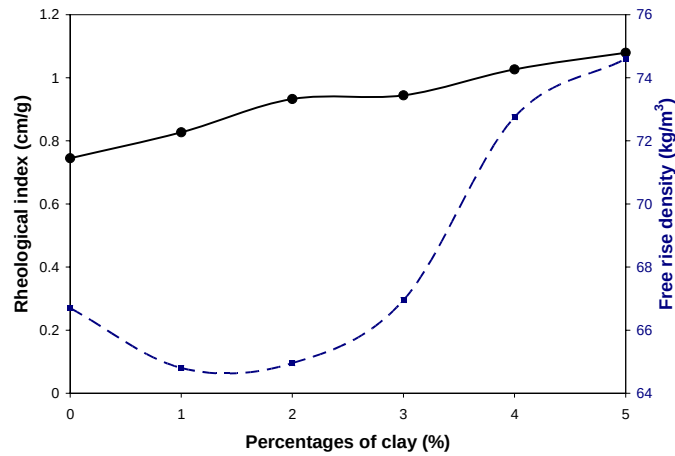


Figure 3: Rheological index and FRD properties

Energy Absorption

Energy absorption is evaluated by calculating the area under the stress-strain graph from compression test. When a foam is loaded, work is done by the force applied to it. Thus, the addition of fillers in production of PU foam is found to increase the work and increase the absorption energy (Figure 4(a)).

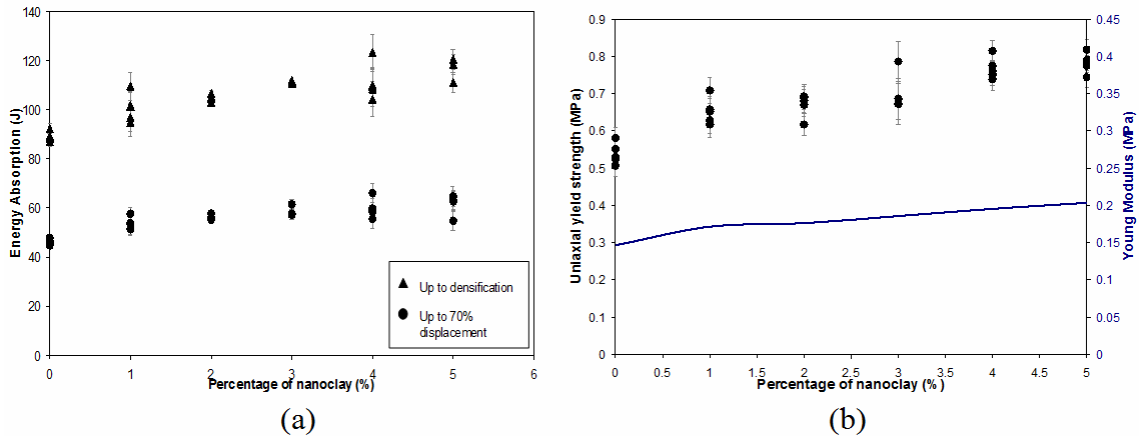


Figure 4 : Effect on additional of nanoclay filler to the value of ; (a) energy absorption, (b) uniaxial yield strength and Young's modulus.

As shown in Figure 4(b), the value of uniaxial yield strength and Young's modulus are also directly proportionate with percentages of nanoclay. It is believed that the increasing in these mechanical properties is also affected by the increasing in density of

the foam as a result of the addition of nanoclay filler.

Sandwich Composites Al-PU Foam

In order to study the effect of skin attachment to PU foam, sandwich composite were prepared and failure mode under flexural testing was investigated. Figure 5 shows the picture of sample after unloading of both PU foam and sandwich composite. Although PU foam is a rigid material, it will not undergo a brittle failure when aluminium skin is attached as shown in Figure 5(b). A sandwich structure consists of three distinct layers (i.e. top skin, core and bottom skin), which are bonded together to form an efficient load carrying assembly. According to Gibson and Ashby [6], the separation of the skins by the core increases the moment inertia of the sandwich panel with little increase in weight, producing an efficient structure for resisting bending and buckling loads.

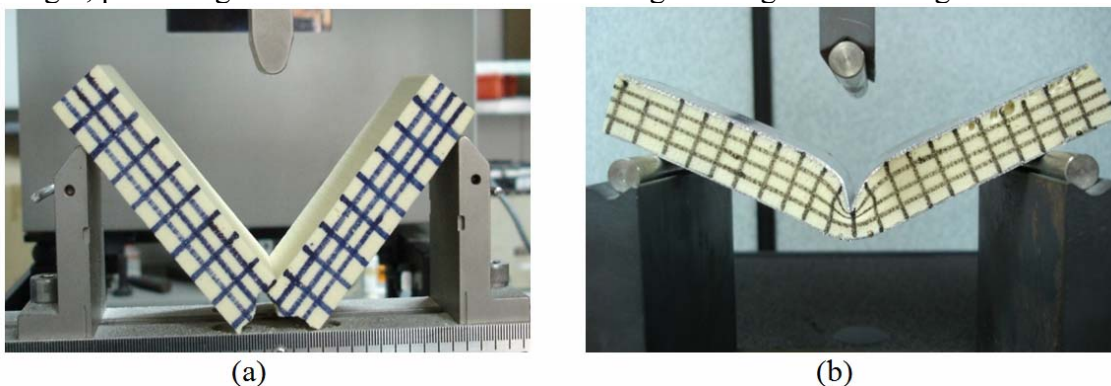


Figure 5 : Photographs of sample after unloading (a)PU foam, (b)Sandwich Al + PU foam.

As shown in Figure 6, flexural stress of sandwich composite is higher than PU foam by almost 346% for sandwich with PU foam without filler as the core. This trend illustrates that addition of skin has improved the mechanical properties compared with PU foam core. This is probably due to the presence of the stiffer skin and relatively weaker core, which causes a uniform shear stress distribution across the thickness of the core. Conversely, when the percentages of filler is increased, flexural stresses for sandwich composite decrease due to increasing in rigidity of PU foam core when the composition of nanoclay added is higher.

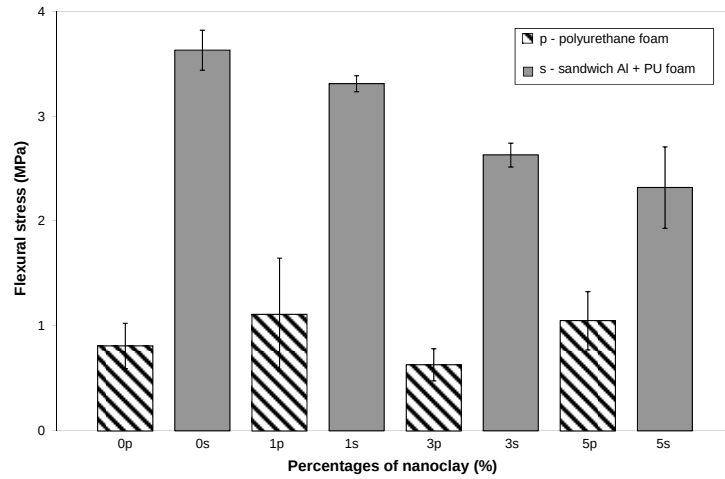


Figure 6: Flexural stress of PU foam and sandwich composite.

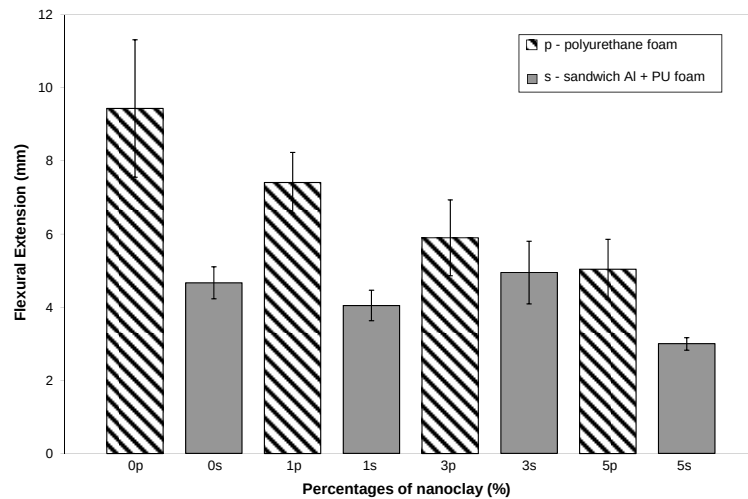


Figure 7 : Flexural extension of PU foam and sandwich composite.

Figure 7 shows the flexural extension of PU foam and sandwich composite. Inversely from flexural stress data, flexural extension of PU foam is higher than sandwich composite. This is probably due to ductility of aluminium skin, which prevent sandwich composite from expanding during flexural testing.

CONCLUSION

The foaming time has increased by almost 133% for Pu-5. The rheological index increases up to 45% with the addition of 5% nanoclay. The Young's modulus of PU

foam was improved up to 0.20MPa. Addition of nanoclay filler in the production of nanocomposite PU foam is found to increase the value of absorption energy and mechanical properties of foam. The flexural stress of sandwich composite structure has increased by almost 346% compared to core (PU foam). However, flexural extension for sandwich composite is lower than the core due to ductility effect of aluminium skin

ACKNOWLEDGEMENTS

The authors would like to thank Degussa/Goldschmidt Chemical Corp, Southern Clay Products and Maskimi Polyol Sdn Bhd for providing materials, Universiti Malaysia Perlis for SLAB fellowship support and Ministry of Science, Technology and Innovation for financial support under IRPA 03-01-02-SF0223

REFERENCES

- [1]. C. Xia, L. James, W. Tomy, M. Christopher (2005) Polyurethane/clay nanocomposites foams: processing, structure and properties. *Polymer* **46**, 775-783
- [2]. F.V.V. de Sousa, R.O. da Mota, J.P. Quintela, M.M. Vieira, I.C.P. Margarit, O.R. Mattos (2007) Characterization of corrosive agents in polyurethane foams for thermal insulation of pipelines. *Electrochimica Acta* doi:10.1016/j.electacta.2006.12.074
- [3]. M. Ionescu. 2005. *Chemistry and technology of polyols for polyurethane*, Rapra Technology Limited, UK
- [4]. K.H. Badri, Z. Othman, and S.H. Ahmad (2004) Rigid polyurethane foams from oil palm resources, *J. of Materials Science* **39**, 5541-5542
- [5]. Wada, A., T. Kawasakit, Y. Minoda, A. Kataoka, S. Tashiro and H. Fukuda (2003) A method to measure shearing modulus of the foamed core for sandwich plates. *J Composite Structures*, Vol 60, pp 385 – 390
- [6]. Gibson, L.J. and M. Ashby, 1998. *Cellular Solids: Structure and Properties* Second Edition, United Kingdom, Cambridge University Press