PROPERTIES OF MODIFIED CALCIUM CARBONATE/NATURAL FIBER FILLED POLYETHYLENE HYBRID FOAM COMPOSITES

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Dedicated to my late dad and mom
I will always missing you

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ABSTRACT

Modified calcium carbonate ($CaCO_3$) filler was analyzed for its thermal, biodegradability and hazardousness of the material to the environment. Then, modified $CaCO_3$/natural fiber filled low density polyethylene (LDPE) hybrid foam composites were produced and characterized for their physical and mechanical properties. Hybrid $CaCO_3$/natural fiber LDPE foam composites were prepared by melt mixing in two roll mill and foaming process was done using hot press at 160 °C with varied amounts of modified $CaCO_3$ filler. Fibers used in this experimental were kenaf, banana pseudostem, coconut coir and pineapple leaf. The amounts of natural fiber were varied from 0 to 40 part per hundred (phr). Biodegradability test using enzymatic and soil burial for modified $CaCO_3$ showed decrease in weight after 30 days of incubation and 49 days (7 Week), respectively. No abnormality showed from animal and terrestrial plant for hazard assessment of modified $CaCO_3$. Sample containing 10 phr of modified $CaCO_3$ filler showed the highest value of flexural strength which was 289.22 MPa. For all samples, increasing the modified $CaCO_3$ filler increased the compressive strength of the foam composite. Density of foam composites increased with increasing the amount of modified $CaCO_3$ filler and all types of fiber loading. The effect of kenaf fiber loading showed 15 phr of fiber represented the highest value of flexural strength which was 1531.30 MPa. Increasing fiber loading into foam composite resulted in decreasing flexural strength and cause lowered the compressive strength. Effects of different types of fiber on foam composites were evaluated. Kenaf showed the highest flexural strength which was 1531.30 MPa and coconut coir fiber showed the highest result which was 51.23 MPa for compressive strength.

ABSTRAK

Pengisi kalsium karbonat ($CaCO_3$) terubahsuai telah dianalisis untuk termal, keterbiodegradasikan dan tahap bahaya bahan kepada alam sekitar. Kemudian, komposit busa polietilena ketumpatan rendah (LDPE) terisi hibrid $CaCO_3$ terubahsuai/gentian semulajadi telah dihasilkan dan dicirikan untuk sifat fizikal dan mekanikalnya. Komposit busa LDPE hibrid $CaCO_3$/gentian semulajadi telah disediakan dengan percampuran leburan menggunakan mesin penggolek berkembar dan proses pembusaan dilakukan menggunakan mesin tekanan panas pada suhu 160 °C dengan kandungan pengisi $CaCO_3$ terubahsuai yang berbeza. Gentian yang digunakan di dalam eksperimen ini adalah kenaf, batang semu pisang, sabut kelapa dan daun nanas. Jumlah gentian semulajadi yang digunakan berubah daripada 0 sehingga 40 bahagian per seratus (phr). Ujian keterbiodegradasikan menggunakan enzim dan ditanam dalam tanah untuk $CaCO_3$ terubahsuai menunjukkan penurunan berat sampel masing-masing setelah 30 hari inkubasi dan 49 hari (7 Minggu). Tiada kelainan ditunjukkan daripada haiwan dan tumbuhan daratan untuk penilaian tahap bahaya daripada pengisi $CaCO_3$ terubahsuai. Sampel mengandungi 10 phr pengisi $CaCO_3$ terubahsuai menunjukkan nilai kekuatan lenturan tertinggi iaitu 289.22 MPa. Untuk semua sampel, peningkatan pengisi $CaCO_3$ terubahsuai telah meningkatkan kekuatan mampatan komposit busa. Ketumpatan komposit busa meningkat apabila jumlah pengisi $CaCO_3$ terubahsuai dan semua jenis gentian semulajadi meningkat. Kesan pemuatan gentian kenaf menunjukkan 15 phr gentian menghasilkan nilai tertinggi bagi kekuatan lenturan iaitu 1531.30 MPa. Peningkatan pemuatan gentian dalam komposit busa menurunkan kekuatan lenturan dan menyebabkan penurunan kekuatan mampatan. Kesan untuk berbagai jenis gentian ke atas komposit busa telah dinilai. Kenaf menunjukkan kekuatan lenturan yang tertinggi 1531.30 MPa dan sabut kelapa menunjukkan nilai tertinggi dalam kekuatan mampatan iaitu 51.23 MPa.

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LIST OF SYMBOLS

%	-	percent
°C	-	degree celcius
°C/min	-	degree celcius per minute
μm	-	micrometre
cm	-	centimetre
cm^{-1}	-	per centimetre
Ef	-	foaming efficiency
Ev	-	foaming effectiveness
g	-	gram
g/cm^3	-	gram per cubic metre
h	-	hour
kV	-	kilovolt
mg	-	milligram
min	-	minute
min^{-1}	-	per minute
ml	-	millilitre
ml/min	-	millilitre per minute
mm	-	millimetre
T_c	-	crystallization temperature
T_m	-	melting temperature
w/v	-	weight per volume
L	-	litre
GPa	-	gigapascal
N	-	newton
MPa	-	megapascal

LIST OF ABBREVIATIONS

ADC	-	Azodicarbonamide
ASTM	-	American standard testing materials
$CaCO_3$	-	Calcium carbonate
CO_2	-	Carbon dioxide
DCP	-	Dicumyl peroxide
DSC	-	Differential scanning calorimetry
EPDM	-	Ethylene propylene diene monomer
EVA	-	Ethylene-vinyl acetate
FTIR	-	Fourier Transform-Infrared
HDPE	-	High density polyethylene
LDPE	-	Low density polyethylene
N_2	-	Nitrogen gas
NaOH	-	Sodium hydroxide
PE	-	Polyethylene
PLA	-	Poly(lactic) acid
PP	-	Polypropylene
PS	-	Polystyrene
PU	-	Polyurethane
PVC	-	Polyvinyl chloride
RIM	-	Reaction-injection molding
SAXS	-	Small-angle X-ray scattering
SEM	-	Scanning electron microscopy
TGA	-	Thermogravimetric analysis
UV	-	Ultraviolet
ZnO	-	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In foam industry, the challenges that encounter were waste disposal (including biodegradability), recycling and economically feasible (Klempner and Sendijarevic, 2004). Due to production of foam that is frequently from petroleum based material, the usage of synthetic resins alone in plastic compounding results in longer time was taken to degrade in landfill. The lack of degradability and compostability lead to the modification of polymer chain of petroleum-based resin as the alternative way to sustain the environmental.

Generally, the addition of calcium carbonate ($CaCO_3$) may decrease the tensile strength, compare with similar quantities of silicates in polymer compound, also, addition of untreated $CaCO_3$ in polymer compound reduces impact strength (Waterman *et al*., 1978). However, calcium carbonate appeared to be an effective additive to enhance the compressive behaviours (Konar *et al*., 2013). This is the most popular mineral filler in plastic industry which are economical and compatible with a wide range of polymer resins.

One option for the replacement of petroleum-based polymers is natural polymers such as native starch, fiber and chitosan. The natural polymers are readily available, inexpensive and biodegradable (Tharanathan, 2003). The attention on natural plant fibers as reinforcement in plastic application to replace synthetic fibers arise as they are renewability, low density and high specific strength (Ochi, 2008). By introducing the kenaf fibers to the foamed composite, the tensile strength will be improved because kenaf fiber is known to have the potential as a reinforcing fiber in thermoplastic composites (Karnani *et al*., 1997). Addition of kenaf fibers gives tendency of foamed composite susceptible to be attack by microorganism during disposal.

Abundantly waste from agriculture and the availability give idea to researchers to develop the usage of natural fibers. The uses of these fibers are driven solely by their environmental attributes and inexpensive nature. Sugarcane bagasse, oil palm empty fruit bunch and banana pseudo-stem are some of the natural cellulosic fibre that widely available in Malaysia. These advantages make us interested to add some value from this waste in polymer foam production. In addition, agricultural wastes can have great commercial application and facilitate extra income to farmers. This works is one of the efforts to utilize the usage of agricultural waste.

Foams can be flexible or rigid and their properties depend on the chemical nature of the polymer, the manufacturing process, the density and the cell morphologies (Biron, 2007). Polyolefin foams are flexible, tough and good resistant to chemical and abrasion. Generally, they posses intermediate mechanical properties between rigid and highly flexible foams. They are known to have good thermal insulation and superior electrical properties (Landrock, 1995). Yu *et al*., (2011) have reported that low-density polyethylene (LDPE) has wide foaming temperature window because the high degree of long chain branching lead to higher macromolecular entanglement density and higher macromolecular chain interactions. This made the cells growth easy to stabilize. Since early 1940s, LDPE foam is produced and become a major commercial foam product with wide application because it outstanding foamability (Yu *et al*., 2011).

1.2 Problem Statements

As an alternative way for cost saving and reduce the consumption of petroleum-based material, inorganic filler such as calcium carbonate has been used in plastic industries. Using $CaCO_3$ alone in compounding may decrease the properties compare to pure resin. The addition of compatibilizer, coupling agent and chemically modification on $CaCO_3$ itself will help good interfacial with polymer matrix and directly contributed to sufficient stress transfer from matrix to fibre. Modified $CaCO_3$ have been used in this research. The modified filler contains 90 % of $CaCO_3$ and consists of alkane, amides, ethylene propylene diene monomer (EPDM), stearic acid and silane.

One of the main problems of LDPE foam is not readily biodegradable. Addition of modified $CaCO_3$ in LDPE foam cannot solve the environmental issue because this inorganic filler will normally undergo carbon mineralization and deterioration if thermal oxidation occurs during compost. Li *et. al*., (2013) said only one weight loss from three or four phase during the decomposition of calcium carbonate at 600 – 850 °C. Natural fibers that abundantly and wide availability and other reachable agro-waste around the world contribute the sustainable technology research. The main purpose to yield lighter composites with lower cost introduced natural fibres compared to high expensive reinforced polymer composites using glass fibre. Common petroleum-based plastics, such as polyethylene (PE) and polypropylene (PP) are used widespread with natural fibres like sisal, hemp, kenaf, and jute (Bledzki and Gassan, 1999). The combination of natural fibers to reinforced PE foam also will help increase bioavailability of the biodegradation in the compound.

Foam performance can be enhanced by fiber reinforcement (Kannan et al., 1981). Fiber length, fiber-volume fraction, fiber content and fiber orientation give great influence in the strength of the foam. Longer fibers were significantly more effective than shorter fiber in improving strength and modulus of foams under tensile and flexural loading. However, long fiber having difficulties during processing and it is a challenge to prevent fiber breakage during the compounding. Lee *et al.*, (2013) has used internal mixer in their research face fiber breakage problem and indirectly reduce the reinforcing capability of the kenaf fiber in polypropylene and poly(butylene succinate) composites. Thus, in this research, two roll mills have been used to compound the LDPE resin with other ingredients.

In this project, the following questions arise and have been identified:

1. What are the properties of modified $CaCO_3$?
2. What are the optimum amount and processing parameter of modified $CaCO_3$ filler compounded with LDPE?
3. What are the physical and mechanical properties of hybrid foam composites with additional of natural fibers?

1.3 Objectives

The aim of this research was to study the properties of hybrid LDPE foam composites using modified $CaCO_3$ and natural fibers. The objectives have been formulated to achieve the research aims such as below:

i. To analyzed the modified $CaCO_3$ filler for its thermal characterization, biodegradability and assessing the hazard of material.

ii. To determine the effects of modified $CaCO_3$ filler and kenaf fiber loading in LDPE foam composite based on density and mechanical test (flexural and compression test).

iii. To determine the effects of different natural fibers on density and mechanical properties.

1.4 Scope of Study

This research is aiming to study the modified $CaCO_3$ filler and the application in commodity plastic as foam composite. In order to achieve the objectives of this research, the procedures taken are followed by the step below:

1. Modified $CaCO_3$ filler characterization.

i) Samples were brought for Fourier Transform-Infrared (FTIR), Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA).

ii) Enzymatic degradation/fungal resistance (ASTM G21) and soil burial test were done to investigate the degradation properties.

iii) Assessment on hazard of material.

2. Foam composite preparation.

i) Optimum amount of modified $CaCO_3$ were determined using different loading, 5, 10, 15, 20, 30, and 40 phr using two rolls mill at temperature of 120 °C for 10 mins.

ii) The sheet of LDPE was brought to hot air by hot press for foaming process using temperature of 160 °C for 15 mins.

iv) Foamed sheet was cut into dimension 100 mm x 20 mm x 30 mm for further testing.

v) Natural fiber hybrid foam composites were prepared with a similar method and the fiber loading varies from 5 to 40 phr.

3. Mechanical properties studies

i) Flexural strength – ASTM D790-10

ii) Compression strength – ASTM D3574

4. Physical properties studies

i) Density - ASTM D1622-09

ii) Moisture Content – ASTM D644

1.5 Significance of Study

LDPE is commodity plastic and well-known having unique strain-hardening characteristics for high degree foam expansion. LDPE foam have many advantages in foam application, unfortunately, poor in biodegradability reduce the interest as people nowadays are compete to create green invention and sustainability.

This study explores the addition of modified $CaCO_3$ in LDPE foam composite with treated agricultural waste fibers. The combination of natural fibers will reduce the problem of biodegradability and cost effectiveness for disposable foam packaging. It is essential to keep the new invention more eco-friendly to environment. Four types of natural cellulosic fibers were used to compare the mechanical strength, physical properties and morphology. Kenaf bast, banana pseudo-stem, coconut coir and pineapple leaf were left to decompose in plantation field can be obtained widely in Malaysia was chosen to turn from landfill waste into useful things.

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