MECHANICAL, THERMAL, TRIBOLOGICAL AND FLAMMABILITY PROPERTIES OF HYBRID SYNTHETIC WOLLASTONITE NANOFIBER/ GRAPHENE OXIDE REINFORCED POLYBUTYLENE TEREPHTHALATE NANOCOMPOSITES

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ABSTRACT

The demand of lightweight materials and fuel economy enhancement has driven the development of polymer nanocomposites. In this study, novel nanocomposites based on polybutylene terephthalate (PBT) and synthetic wollastonite nanofibers (SWN) were developed. SWN were synthesized via hydrothermal reaction under different reaction mediums and temperatures, followed by calcination. The products were confirmed as high-purity wollastonite in nanosize, with either granular or fiber form. SWN with highest aspect ratio of 16.3 produced under reaction medium mixture of 20 v/v% ethanol, 80 v/v% water and reaction temperature of 200 ºC, was used in PBT nanocomposites. The effect of SWN contents on the mechanical, thermal, tribological and flammability properties of the PBT/SWN nanocomposites were studied. Test samples were fabricated via melt compounding method. The addition of SWN into PBT resulted in the maximum increment of tensile strength (6%) and Young's modulus (13%) due to its reinforcing effect and good interactions with PBT matrix via hydrogen bonding. However, elongation at break and impact strength demonstrated decreasing trends with increasing SWN contents. PBT reinforced with 1.0 phr SWN exhibited the best combination of stiffness and toughness. A significant increase in wear resistance (73%) was observed at the same SWN content, whereas friction coefficient decreased with increasing SWN contents. The incorporation of SWN had increased the thermal properties and the thermal stabilities of the nanocomposites, and simultaneously suppressed the peak rate of heat release and the rate of production of smoke and toxic gases. PBT/SWN 1.0 nanocomposite with the most balanced properties was used to compare with the natural wollastonite (NW)- and graphene oxide (GO)-reinforced PBT composites at the same content. Similar to SWN, hydrogen bonds were formed between the NW filler-PBT matrix interface. However, due to the larger surface area possessed by SWN, its nanocomposite exhibited higher tensile strength, Young's modulus and wear resistance than that of NW. GO demonstrated poor interfacial adhesion with PBT matrix, thus had inferior mechanical properties and wear resistance. Nonetheless, PBT/GO 1.0 nanocomposite had the best anti-friction performance among the PBT composites due to the lubricating ability of GO. All fillers were able to improve the thermal and the flammability properties of PBT, where the degradation temperatures were significantly increased for PBT/SWN 1.0 and PBT/GO 1.0 nanocomposites by 9 - 14 ºC. PBT/SWN 1.0 nanocomposite was also used as the base material to fabricate PBT/SWN/GO hybrid nanocomposites with 0.5 - 2.0 phr GO contents. By increasing the GO contents in hybrid nanocomposites displayed further improvement in Young's modulus (16%) due to the better dispersion of GO nanosheets. However, due to the lacking of interfacial adhesion between GO and PBT matrix, the tensile strength, elongation at break, impact strength and wear resistance of hybrid nanocomposites were inferior than PBT/SWN 1.0 nanocomposite. The addition of 1.5 phr GO had attained the lowest friction coefficient with 34% reduction from that of neat PBT. Hybridization of SWN and GO further promoted crystallization, delayed the thermal degradation and improved the flame retardancy of hybrid nanocomposites. Overall study showed that the multifunctional PBT nanocomposites based on SWN and GO have great potential for lightweight structural components, thereby expanding the applications of PBT in automotive industry.

ABSTRAK

Permintaan terhadap bahan yang ringan dan penjimatan bahan api telah memacu pembangunan nanokomposit polimer. Nanokomposit berasaskan polibutilena tereftalat (PBT) dan nanogentian wollastonite sintetik (SWN) dihasilkan dalam kajian ini. SWN telah disintesis melalui tindak balas hidroterma dalam pelarut dan suhu tindak balas yang berbeza, diikuti dengan pengkalsinan. Produk terhasil telah disahkan sebagai wollastonite yang berketulenan tinggi bersaiz nano, berbentuk butiran atau gentian. SWN yang mempunyai nisbah aspek tertinggi iaitu 16.3 digunakan sebagai pengisi nano dalam nanokomposit PBT, ianya dihasilkan secara pukal dalam campuran pelarut 20 v/v% etanol, 80 v/v% air dan suhu 200 ºC. Kesan kandungan SWN pada sifat mekanikal, haba, tribologi dan kebolehbakaran nanokomposit PBT/SWN telah dikaji. Sampel ujian telah dihasilkan melalui kaedah penyebatian lebur. Penambahan SWN meningkatkan kekuatan tegangan (6%) dan modulus Young (13%) disebabkan oleh kesan pengukuhan SWN dan interaksi yang baik dengan PBT melalui ikatan hidrogen. Walau bagaimanapun, pemanjangan takat putus dan kekuatan hentaman menurun dengan peningkatan kandungan SWN. PBT diisi dengan 1.0 phr SWN mempamerkan gabungan kekakuan dan keliatan yang terbaik. Peningkatan ketara dalam rintangan haus (73%) dapat dilihat pada 1.0 phr SWN, manakala pekali geseran menurun dengan peningkatan kandungan SWN. Kehadiran SWN meningkatkan sifat terma dan kestabilan terma dan menurunkan kemuncak kadar pelepasan haba dan kadar pengeluaran asap dan gas toksik. PBT/SWN 1.0 mempunyai sifat yang paling seimbang telah dibandingkan dengan komposit yang berasaskan wollastonite semula jadi (NW) dan grafin oksida (GO) dengan kandungan yang sama. Sama seperti SWN, ikatan hidrogen wujud di antara permukaan PBT dan NW. Nanokompositnya mempamerkan kekuatan modulus Young dan rintangan haus yang lebih tinggi daripada NW disebabkan oleh luas permukaan yang lebih besar yang dimiliki oleh SWN. GO menunjukkan perekatan yang lemah dengan matrik PBT menyebabkan sifat mekanikal dan rintangan haus yang lebih rendah. Namun begitu, nanokomposit PBT/GO 1.0 mempunyai prestasi anti-geseran yang terbaik di kalangan komposit PBT kerana keupayaan pelinciran GO. Semua pengisi berupaya untuk meningkatkan sifat terma dan kebolehbakaran PBT yang mana suhu perosotan telah meningkat dengan ketara untuk nanokomposit PBT/SWN 1.0 dan PBT/GO 1.0 sebanyak 9 ºC - 14 ºC. Oleh itu, nanokomposit PBT/SWN 1.0 telah digunakan sebagai bahan asas untuk menghasilkan nanokomposit hibrid PBT/SWN/GO dengan 0.5 - 2.0 phr kandungan GO. Peningkatan kandungan GO dalam nanokomposit hibrid menunjukkan penambahbaikan dalam modulus Young (16%) disebabkan oleh serakan nanopengisi GO yang lebih baik. Walau bagaimanapun, disebabkan kekurangan lekatan antara GO dan PBT, kekuatan tegangan, pemanjangan semasa putus, kekuatan hentaman dan rintangan haus nanokomposit hibrid adalah lebih rendah daripada nanokomposit PBT/SWN 1.0. Penambahan 1.5 phr GO mencapai pekali geseran terendah dengan pengurangan 34% daripada PBT. Hibridisasi SWN dan GO menggalakkan lagi penghabluran, melambatkan degradasi terma dan meningkatkan rencat nyalaan nanokomposit hibrid. Kajian keseluruhan menunjukkan bahawa nanokomposit PBT berdasarkan SWN dan GO mempunyai potensi yang besar dalam komponen struktur ringan serta pengembangan aplikasi PBT dalam industri automotif.

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LIST OF ABBREVIATIONS

Al_2O_3 - Aluminium oxide
ASTM - American Standard Testing Method
B_2O_3 - Boron trioxide
C - Carbon
Ca - Calcium
CAGR - Compound annual growth rate
$CaSiO_3$ - Calcium metasilicate
$Ca(NO_3)_2.4H_2O$ - Calcium nitrate tetrahydrate
$Ca(OH)_2$ - Calcium hydroxide
$Ca_5(Si_6O_{16})(OH)_2.4H_2O$ - Tobermorite
$Ca_6(Si_6O_{17})(OH)_2$ - Xonotlite
CNTs - Carbon Nanotubes
CO - Carbon monoxide
CO_2 - Carbon dioxide
CO_3^{2-} - Carbonate ion
CTAB - Cetyltrimethylammonium bromide
D - Diameter
DSC - Differential scanning calorimeter
EDS - Energy dispersive X-ray spectroscope
FESEM - Field emission scanning electron microscope
Fe_2O_3 - Iron (III) oxide
FIGRA - Fire growth rate
FPI - Fire performance index
FTIR - Fourier-transform infrared spectrometer
GO - Graphene oxide
GO-OH - Multi-hydroxyl-functionalized graphene oxide
H - Hydrogen
HDPE - High density polyethylene
HRR - Heat release rate
H_2O - Water

ICDD	-	International center for diffraction data
KBr	-	Potassium Bromide
L	-	Length
LOI	-	Limiting oxygen index
MEGONS	-	Microwave exfoliated graphite oxide nanosheets
MMT	-	Montmorillonite
Na_2O	-	Sodium oxide
$Na_2SiO_3.5H_2O$	-	Sodium meta-silicate-pentahydrate
NPCC	-	Nano-precipitated calcium carbonate
NW	-	Natural wollastonite
O	-	Oxygen
OH	-	Hydroxyl
PA	-	Polyamide
PAN	-	Polyacrylonitrile
PBT	-	Polybutylene terephthalate
PBT-g-AA	-	Acrylic acid-grafted polybutylene terephthalate
PBS	-	Poly(butylene succinate)
PC	-	Polycarbonate
PCOP	-	Peak of carbon monoxide production
PCO_2P	-	Peak of carbon dioxide production
PEGMA	-	Poly (ethylene glycol) methacrylate
PET	-	Polyethylene terephthalate
PHRR	-	Peak of heat release rate
POD	-	Pin-on-disc
POM	-	Polyoxymethylene
PP	-	Polypropylene
PPcoE	-	Polypropylene-ethylene copolymer
PSPR	-	Peak of smoke production rate
rGO	-	Reduced graphene oxide
rPC	-	Recycled polycarbonate
rPET	-	Recycled polyethylene terephthalate
Sb_2O_3	-	Antimony trioxide
Si	-	Silicon
SiC	-	Silicon carbide

SPR	-	Smoke production rate
SSR	-	Solid-state reaction
SWN	-	Synthetic wollastonite nanofibers
TEOS	-	Tetraethyl orthosilicate
TGA	-	Thermogravimetric analyzer
TTI	-	Time to ignition
UHMWPE	-	Ultra-high-molecular weight polyethylene
XRD	-	X-ray diffractometer
1D	-	One-dimensional
2D	-	Two-dimensional
3D	-	Three-dimensional

LIST OF SYMBOLS

cc - Cubic centimeter
cm - Centimeter
d - Sliding distance
F_N - Normal applied load
g or gm - Gram
GPa - Giga Pascal
g/cm^3 - Gram per centimeter cube
g/s - Gram per second
h - Hour
Ibs - Pounds weight
J/g - Joule per gram
J/m - Joule per meter
kN - Kilonewton
kV - Kilovolt
kW/m^2 - Kilowatt per meter square
kW/m^2·s - Kilowatt per meter square·second
L/D - Aspect ratio length over diameter
M - Molar concentration
m - Meter
mA - Milliampere
mg - Milligram
min - Minute
MJ/m^3 - Mega joule per meter cube
mm - Millimeter
mm^3 - Millimeter cube
mm^3/N·m - Millimeter cubic per newton·meter
MPa - Mega Pascal
mW - Milliwatt
m/s - Meter per second
m^2.s/kW - Meter square·second per kilowatt

m^2/s - Meter square per second

m^2/g - Meter square per gram

N - Newton

nm - Nanometer

phr - Parts per hundred resin

rpm - Revolutions per minute

s - Second

T_c - Crystallization temperature

$T_{c,o}$ - Onset crystallization temperature

T_{endset} - End set degradation temperature

T_g - Glass transition temperature

T_m - Melting temperature

T_{max} - Temperature at maximum degradation rate

T_{m1} - First melting peak in second heating

T_{m2} - Second melting peak in second heating

T_{onset} - Onset degradation temperature

TPa - Tera Pascal

T_{20} - Temperature at 20% mass loss

T_{50} - Temperature at 50% mass loss

vol% - Volume percent

w_{PBT} - Mass fraction of PBT in nanocomposites

wt% - Weight percentage

W_s - Specific wear rate

W/m.K - Watts per meter-Kelvin

X_c - Degree of crystallinity

X_{c2} - Degree of crystallinity for second heating

α - Alpha

β - Beta

µm - Micrometer

μ - Friction coefficient

μm^2 - Micrometer square

°C - Degree Celsius

° - Degree

ΔH_c - Crystallization enthalpy

ΔH_m - Melting enthalpy

ΔH_{m2} - Melting enthalpy for second heating

ΔH°_m - Melting enthalpy of 100% crystalline PBT

ΔV - Volume difference

% - Percent or Percentage

% v/v - Percentage of volume per volume

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Due to the limited natural resources and stricter environmental regulations, fuel efficiency has become one of the most important featured in automotive design. Developing new materials and re-designing of the existing one, particularly the advanced plastics and polymer composites have driven the advancements in automotive industry. Engineering thermoplastics are playing vital roles in industries such as automotive, construction and electrical/electronic industries (Sanusi, Benelfellah, and Aït Hocine, 2020). In automotive industry, they are replacing materials such as metals and glasses to reduce weight and improve fuel economy (Ramanjaneyulu et al., 2017; Ibeh, 2011). Usage of plastics and polymer composites as a substitute for heavier materials can lead to an overall 10% weight reduction with a 3 – 7% improvement in fuel efficiency (Miller et al., 2014). Figure 1.1 shows the average material composition for a passenger vehicle based on year, indicating the increasing mass percentage for plastics from 6% to 18% (Rouilloux et al., 2012; Miller et al., 2014).

Polymer composite consist of two or more components, with filler materials as the most important additives to polymers. Polymer nanocomposite is an alternative to the conventional polymer composites, at which the filler material used is in nanoscale. Nanoscale fillers have at least one dimension of 100 nm or lesser and vary essentially from isotropic to highly anisotropic morphologies (Fu et al., 2019; Szeluga, Kumanek, and Trzebicka, 2015). The growth of polymer composite in various applications is due to their outstanding strength to weight ratio and cost to performance ratio when compared to that of metals. In the last two decades, the development of polymer nanocomposite has replaced the traditional polymer composite. Since the discovery of the excellent performance of organophilic clay reinforced polyamide (PA) 6 by Toyota Central Research Laboratories, Japan in 1990, polymer nanocomposite has received much attention as very little amount of nano-filler can significantly improve thermal and mechanical properties of the polymer matrix (Sanusi et al., 2020). Nanoscale fillers provide relatively larger surface interactions with the polymer matrix, leading to the properties enhancement of polymer matrix at very low filler content (Fu et al., 2019; Sanusi et al., 2020). Besides its effectiveness, polymer nanocomposites stand out as one of the most promising technologies because its processing and manufacturing technique is similar to that of conventional polymer composites. Hybrid nanocomposite is another focus in polymer industry as the need for high performance polymer composites is increasing. Hybrid polymer composites are those systems in which one type of filler material is introduced into a mixture of different matrixes, two or more filler materials are introduced into a single matrix, or the combination of the two approaches (Szeluga et al., 2015; Fu et al., 2019). To achieve desired properties, polymer nanocomposites with multi-type fillers have been developed (Ding et al., 2019; Phutfak and Larpkasemsuk, 2021). By producing a hybrid polymer composite, it may be possible to create a material with combined advantages of the individual components (Fu et al., 2019).

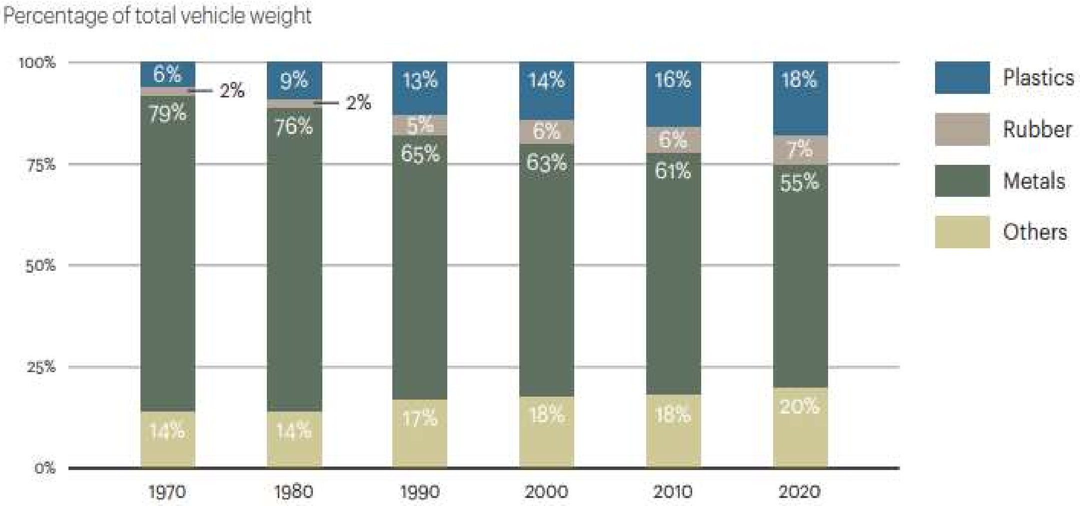

Figure 1.1 Average material composition of a passenger vehicle in Europe (Rouilloux et al., 2012)

Polybutylene terephthalate (PBT) is the engineering thermoplastic counterpart of polyethylene terephthalate (PET) commercialized in 1969 (Ibeh, 2011). It is one of the raising engineering polymers. The global market for PBT was estimated at 1.3 Million Metric Tons in the year 2022, and is projected to reach 1.5 Million Metric Tons by year 2026, growing at a compound annual growth rate (CAGR) of 4.7% (Global Industry Analysts, 2022). According to the world market report by Global Industry Analysts in 2022, the largest end-use market of PBT is in automotive and followed by the electronics and electrical sector. Owning to the increasing demands for lightweight, cost-effective and low-maintenance materials, PBT is projected to grow at 5.1% CAGR in automotive segment for the next four years. PBT is commonly applied as exterior and interior automotive parts such as fuel system components, mirror housings and ignition system components. It gained footing in the market with its exclusive properties in dimensional stability, chemical resistance, dielectric properties, and heat resistance. However, due to its relatively inferior strength, rigidity, thermal stability, friction performance and flammability, PBT is often added with fillers to form polymer composites and nanocomposites (Chow, 2015).

Wollastonite is a biocompatible mineral filler with many unique characteristics and it can act as reinforcing agent and functional filler in polymer materials. It is commonly known as calcium metasilicate, $CaSiO_3$ and has acicular crystal shape with high aspect ratio, high hardness and good nucleation ability (Švab et al., 2009; Panin et al., 2020). With this, it has become a common alternative to fiberglass in enhancing the properties of plastics, construction materials and ceramics, as it provides characteristics comparable to those of fiberglass (Panin et al., 2020). Wollastonite is often being used as reinforcing filler in polymers such as polypropylene (PP) (Ding et al., 2019; Luyt et al., 2009), ultra-high-molecular weight polyethylene (UHMWPE) (Danilova et al., 2021; Panin et al., 2020) and PBT (Deshmukh et al., 2022; Deshmukh et al., 2011a). Wollastonite can be obtained naturally or synthesized through chemical reactions. Commercially available wollastonite is naturally obtained from ore through the beneficiation process. Synthetic wollastonite can be found in the market as well, normally being produced through solid-state reaction (SSR). The synthetic wollastonite with controllable quality is preferred as the size, shape and purity of the filler has significant effect on the properties enhancement of polymer composites (Ding et al., 2019; Tong et al., 2006).

Besides the properties of the polymer matrix and fillers, the morphology and size can directly determine the overall properties of the nanocomposite materials (Ding et al., 2019; Tong et al., 2006). In an attempt towards developing high-performance polymeric nanocomposites, combination of two dissimilar but distinctive nanofillers is being introduced into polymer matrix to produce ternary nanocomposites. Also, hybridization of nanofillers provides effective means of ensuring dispersibility of certain nanofiller using other nanofiller (Fu et al., 2019). Thus, graphene oxide (GO) is chosen as the second nanofiller to be incorporated. GO is one of the graphene derivatives. Despite of its natural abundancy, graphene and its derivatives have only recently merged as functional fillers. In recent years, development of graphene-based polymer nanocomposites attracted much attentions. GO is normally produced from graphite through a series of oxidation and exfoliation. Similar to graphene, it is in two-dimensional with planar honeycomb lattice. Its surface area can be greater than 100 μm^2 and its thickness can be less than 1 nm. Due to the presence of oxygen functionalities, it can disperse easily in water, organic solvents and different hydrophilic matrixes. Moreover, GO has exceptional ability in improving mechanical, thermal and friction performances of polymers (Bian et al., 2013; Yetgin, 2020).

1.2 Problem Statement

PBT is one of the most commonly used plastics for automotive components and electrical/electronic appliances such as under-body and exterior body parts of automotive and electronics housings, due to its excellent comprehensive performance. Compared with PA, PBT has better dimensional stability due to its negligible moisture absorption. PBT is more suitable to be used for injection molding than PET due to its lower melt viscosity, good moldability and more rapid crystallization rate. Despite the superior properties of PBT, there are some drawbacks that need to be addressed. PBT has relatively lower tensile strength and tensile modulus when compare with other engineering materials such as PET, PA and polyoxymethylene (POM). It has poorer thermal stability compared to PET and PA. PBT has relatively better tribology properties compared to PET, polycarbonate (PC) and phenolics but inferior compared to PA and POM. These limit its usage especially when the parts are subjected to continuous wear. PBT, PET and PA are relatively flame retardant than POM. However, PBT burns rapidly and produces large amount of smoke and toxic gas. Hence, enhancement in mechanical, thermal, tribological and flammability properties of PBT is vital in order to compete with other engineering polymers or polymer composites for more advance applications in automotive industry.

With this, current work fabricated a novel PBT nanocomposite by incorporating the synthetic wollastonite nanofibers (SWN) to simultaneously enhance the overall functional properties. Wollastonite is one of the most popular and effective functional filler used in polymer composites to achieve better mechanical, thermal, wear resistance and flammability properties (Danilova et al., 2021; Deshmukh et al., 2022; Chaiwutthinan et al., 2019). Natural wollastonite (NW) with acicular shape in broad range of micron-size distribution were commonly used. However, NW can hardly achieve high purity with its non-environmental-friendly beneficiation process. Also, extra processing steps are need to obtain the desired size and shape of NW. Along with the shift towards more sustainable and nanotechnological driven, the synthesis of nano-size wollastonite through various chemical route are receiving significant attention (Raju et al., 2022; Lin et al., 2007). Compared to the large amount of organic solvent required in the beneficiation process of NW ore, chemical routes used to synthesis synthetic wollastonite often use little or no template or organic solvent. Another advantage of synthetic wollastonite over NW is their high quality in terms of purity, as well as their tunable shape and size. Great interest recently arose in this area of study regarding the effect of synthesis parameters on the shape and size of synthetic wollastonite (Xu et al., 2018; Wu et al., 2013). Up to the author's knowledge, only one study reported the effect of reaction medium on the shape of calcium silicate system (Wang et al., 2005). However, the study is not comprehensive. No previous work reported the effect of reaction medium on the size, shape and aspect ratio of synthetic wollastonite. Work investigating the effect of reaction parameters on the aspect ratio of wollastonite produced is very limited (Zhu and Sohn, 2012).

The development of SWN-reinforced PBT nanocomposites in this study was also driven by the perpetual pursuit of lightweight materials for better fuel economy in automotive industry. Synthetic wollastonite has the advantages over NW in terms of its more environmental-friendly synthesis route, high quality and tunable shape and size. Thus, the usage of synthetic wollastonite is getting more interests to be used as nanofillers in woods, wood plastic composites and polymer nanocomposites. To the best of the author’s knowledge, there are very limited work on nano-size synthetic wollastonite-reinforced polymer nanocomposites (Danilova et al., 2021; Luyt et al., 2009; Chatterjee, Khobragade, and Mishra, 2015). Thermoplastics reinforced with synthetic wollastonite of different shapes had been fabricated via melt blending method (Danilova et al., 2021; Luyt et al., 2009). However, no literature reported on the usage of nanostructure synthetic wollastonite in PBT matrix. Other than the advantages of synthetic wollastonite over NW mentioned before, micro size wollastonite as compared to its nano size counterpart gives inferior performance when being used as fillers in advanced materials. Filler size contributed to significant differences in performance enhancement of polymer composites, including mechanical, thermal and tribological properties (Ding et al., 2019; Tong et al., 2006). However, very limited paper reported on the comparative aspects of micro-fillers and nano-fillers on the properties enhancement of polymer composites (Sharma et al., 2015). Moreover, considering the geometry of the particle as an important factor affecting the properties of polymer composites, the effect of SWN, NW and GO of different sizes, shapes and types on the mechanical, thermal, tribological and flammability properties of PBT composites is worth investigating.

Nano-size wollastonite was proven to enhance the mechanical properties and wear performance in reinforced-thermoplastics (Danilova et al., 2021; Luyt et al., 2009), while GO is superior in enhancing the tribological properties, specifically in lubricating the sliding of polymer materials (Bastiurea, Dima, and Andrei, 2018; Yetgin, 2020). Therefore, this research hybridized SWN which is expected to enhance the mechanical properties and GO which is responsible for tribological properties enhancement into PBT matrix to produce PBT/SWN/GO hybrid nanocomposites with balance mechanical, thermal, tribological and flammability properties to offer better alternatives in automotive industry. Work concerning the mechanical, thermal, tribological and flammability properties of hybrid SWN/GO reinforced PBT nanocomposites has not been reported in literature.

1.3 Objectives of Study

The main objective of this research is to develop a new nanocomposite material, hybrid SWN/GO reinforced PBT nanocomposites, which has enhanced performance in terms of mechanical, thermal, tribological and flammability properties. The specific objectives of the research are described as follows.

1. To determine the effect of synthesis parameters (reaction medium and the reaction temperature) on the aspect ratio of the SWN produced.
2. To investigate the effect of SWN content on mechanical, thermal, tribological and flammability properties of the PBT/SWN nanocomposites.
3. To compare the effect of SWN, NW and GO on mechanical, thermal, tribological and flammability properties of the PBT-based composites.
4. To investigate the effect of GO content on mechanical, thermal, tribological and flammability properties of the hybrid SWN/GO reinforced PBT nanocomposites.

1.4 Scope of Study

Based on the objectives, the following has been identified as the scopes of study.

1. SWN were synthesized through a simple hydrothermal method using analytical grade calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$) and sodium meta-silicate-pentahydrate ($Na_2SiO_3 \cdot 5H_2O$) as the reagents. The ethanol composition in reaction medium (0% v/v to 80% v/v) and the reaction temperature (160 °C to

240 °C) were varied to study their effect on the aspect ratio of SWN. All powders produced were characterized using x-ray diffractometer (XRD), fourier transform infrared spectrometer (FTIR), thermogravimetric analyzer (TGA) and field emission scanning electron microscope (FESEM).

2. The preparation of PBT/SWN nanocomposites with different SWN content (0 phr to 3.0 phr) were done by using melt compounding. The structural and morphological characterization of neat PBT and PBT/SWN nanocomposites were studied using FTIR, FESEM and energy dispersive X-ray spectroscope (EDS). The effect of SWN content on the mechanical properties of PBT/SWN nanocomposites were determined by conducting tensile test (ASTM D638) and notched Izod impact test (ASTM D256). Thermal properties were determined using differential scanning calorimeter (DSC) and TGA. Meanwhile, the friction and wear performance were determined using a pin-on-disc (POD) tribo test machine (ASTM G99). Lastly, cone calorimetry (ISO 5660) was used to determine the flammability properties of PBT/SWN nanocomposites.
3. SWN content which exhibited the most balanced functional properties were identified from the PBT/SWN nanocomposites. Similar filler content was used to prepare PBT/NW composite and PBT/GO nanocomposite, via the similar fabrication method with that of PBT/SWN nanocomposites. The effect of filler size and shape on the properties of PBT composites was studied. For comparison purpose, the structural, morphological, mechanical, thermal, tribological and flammability properties of PBT/NW composite and PBT/GO nanocomposite were determined by conducting similar testing.
4. In order to investigate the effect of GO content on mechanical, thermal, tribological and flammability properties of the PBT/SWN/GO hybrid nanocomposites, SWN content was remined constant, while GO content was varied from 0.5 phr to 2.0 phr. Similar fabrication method and testing methods were used in preparing and characterizing PBT/SWN/GO hybrid nanocomposites.

1.5 Significance of Study

This study developed PBT/SWN nanocomposites and PBT/SWN/GO hybrid nanocomposites, which have better mechanical, thermal, tribological and flammability properties than PBT. The mechanical, thermal, wear resistance and flammability properties of neat PBT were enhanced with the inclusion of SWN, while the hybridization of SWN and GO further exhibited synergistic effect in improving the anti-friction performance of the PBT/SWN/GO hybrid nanocomposites. The hybridization of SWN and GO showed more homogenous distribution of GO nanosheets in the PBT matrix, resulting in overall improvement in material properties. This research established the suitable polymer matrix to filler ratios for PBT/SWN nanocomposite and PBT/SWN/GO hybrid nanocomposites in order to achieve the best-balanced performance in mechanical, thermal, tribological and flammability properties. Therefore, lightweight PBT/SWN/GO hybrid nanocomposite with relatively superior properties was fabricated. It explores the potential applications of PBT/SWN/GO nanocomposites in automotive industry in order to replace other materials such as metal which has high density, high-performance polymers which are more expensive and commodity polymers which have inferior performance, for the purpose of safety and better fuel efficiency. The applications of PBT in automotive industry can be extended especially to those parts that require good tribological properties, such as bushings, bearings and gears.

1.6 Thesis Structure

In this research, SWN with different sizes and shapes were successfully synthesized through a simple hydrothermal reaction. SWN with the highest aspect ratio produced from the tailored reaction medium and temperature was incorporated into PBT matrix to enhance the properties. It was further hybridized with GO to formed PBT/SWN/GO hybrid nanocomposites with better properties. Specifically, this research focuses on the mechanical, thermal, tribological and flammability properties of the neat PBT and its composites, at which these properties are taken seriously in automotive industries. This thesis is divided into eight chapters as follows:

- The first chapter provides an overview of the research conducted. It covers the research background, the problem that driven the study, objectives, scopes and the significance of the study.
- The second chapter discussed and evaluated the previous related research reported in the literature to identify the research gaps.
- The third chapter outlined the materials, research design and methods used in this study.
- The fourth chapter reported the results and detailed discussion on the effect of reaction medium and temperature on the aspect ratio of the SWN synthesized.
- The fifth chapter described the variations in mechanical, thermal, tribological and flammability properties of PBT/SWN nanocomposite as a function of SWN content.
- The sixth chapter compared the mechanical, thermal, tribological and flammability properties of PBT/SWN nanocomposite, PBT/NW composite and PBT/GO nanocomposites.
- The seventh chapter analyzed and discussed the effect of GO content on the properties performances of PBT/SWN/GO hybrid nanocomposites.
- The eighth chapter highlighted the findings on the research, assessed the achievement of research objectives and suggested the future research focuses.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Chan J.X.**, Wong J.F., Hassan A., Othman N., Abd Razak J., Nirmal U., Hashim S., Ching Y.C., Yunos M.Z., Yahaya R., & Gunathilake T.M.S.U. (2022). Synthetic wollastonite nanofiber for polybutylene terephthalate nanocomposite: Mechanical, thermal, tribological and flammability properties. *Polymer, 256*, 125259. https://doi.org/10.1016/j.polymer.2022.125259. **(Q1 Journal, IF: 4.432)**

2. **Chan, J.X.**, Wong, J.F., Petrů, M., Hassan, A., Nirmal, U., Othman, N., & Ilyas, R.A. (2021). Effect of nanofillers on tribological properties of polymer nanocomposites: A review on recent development. *Polymers*, *13*(17), 2867. https://doi.org/10.3390/polym13172867. **(Q1, IF: 4.967)**

3. **Chan, J.X.**, Wong, J.F., Hassan, A., Shrivastava, N. K., Mohamad, Z., & Othman, N. (2020). Green hydrothermal synthesis of high aspect ratio wollastonite nanofibers: Effects of reaction medium, temperature and time. *Ceramics International, 46,* 22624-22634. https://doi.org/10.1016/j.ceramint.2020.06.025. **(Q1 Journal, IF:5.532)**

4. **Chan, J.X.**, Wong, J.F., Hassan, A., Mohamad, Z., & Othman, N. (2020). Mechanical properties of wollastonite reinforced thermoplastic composites: A review. *Polymer Composites*, *41*, 395– 429. https://doi.org/10.1002/pc.25403. **(Q2 Journal, IF:3.531)**

5. Wong J.F., **Chan J.X.**, Hassan A., Mohamad Z., Hashim S., Abd Razak J., Ching Y.C., Yunos Z., & Yahaya R. (2022). Use of synthetic wollastonite nanofibers in enhancing mechanical, thermal, and flammability properties of polyoxymethylene nanocomposites. Polymer Composites, (Early View). https://doi.org/10.1002/pc.26902. **(Q2 Journal, IF:3.531)**

6. Wong, J.F., **Chan, J.X.**, Hassan, A., Mohamad, Z., & Othman, N. (2021). Thermal and flammability properties of wollastonite-filled thermoplastic

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7. **Chan J.X.**, Wong J.F., Hassan A., Othman N., Abd Razak J., Nirmal U., Hashim S., Ching Y.C., Yunos M.Z., Yahaya R., & Gunathilake T.M.S.U. Mechanical, thermal, tribological and flammability properties of polybutylene terephthalate composites: Comparing the effects of synthetic wollastonite nanofibers, natural wollastonite and graphene oxide. *Journal of Applied Polymer Science.* **(Q2 Journal, IF: 3.057)** (Accepted with minor correction)

Non-indexed Conference Proceedings

1. **Chan, J.X.**, Wong, J.F., Hassan, A., Othman, N., & Mohamad, Z. (2019). Effect of Hydrothermal Reaction Parameters on The Aspect Ratio of Synthetic Wollastonite Nanofibers. In *7th International Conference and Workshop on Basic and Applied Sciences (ICOWOBAS 2019)* (pp 133).
2. **Chan, J.X.**, Wong, J.F., Hassan, A., Othman, N., Razak, J.A., & Ching, Y.C. (2021). Mechanical Properties and Thermogravimetric Analysis of Synthetic Wollastonite Nanofibers Reinforced Polybutylene Terephthalate Nanocomposites. In *Advanced Nano Materials 2021 (ANM 2021).*