RICE HUSK DERIVED SILICA AEROGEL IN UNSATURATED POLYESTER COMPOSITES

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

School of Mechanical Engineering
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FEBRUARY 2020

DEDICATION

This thesis is dedicated to those who seek knowledge for the sake of knowledge. The author hopes pray for forgiveness, good things in this life and hereafter, if the reader found this thesis useful.

ACKNOWLEDGEMENT

The best of all creation, our Master, Prophet Muhammad, peace and blessings be upon him and his family, said, "He who does not thank people, does not thank Allah" as mentioned in a sahih hadith.

Here, of course, I want to take the opportunity to express my utmost sincere gratitude to all of these important people who inspired me to believe in myself, to push a little harder, and be proud of my goals...

First of all, I would like to thank my supervisor, Assoc. Prof. Dr. Muhamad Azizi Mat Yajid for his support throughout my study. He has been my supervisor for quite a long time, almost 10 years since my Master study, a lot of unforgettable memories...

I would also like to thank my co-supervisor, Prof Dr. Mohd Hasbullah Idris for his motivation and useful advices. Not forgotten, Dato' Prof. Dr. Halimaton Hamdan for the trust given. I've learned quite a lot, over the years, during my research works in her lab.

I wish to thank to Mr Azam from Universiti Sains Malaysia, Mr Shaharunizam from Universiti Malaysia Pahang and Mr Anas from Universiti Teknikal Melaka for their kind cooperation in helping me testing samples and using their instruments.

This study was made possible by financial support from MOE (formerly MOHE) and MESTECC (formerly MOSTI) through research grants and scholarship under MyBrain15 initiatives.

Last, and certainly not least, I'm vastly indebted to my mom and dad which I could never repay. O' Allah! My Lord, have mercy on them, as they raised me when I was a child! Verily, their loves and prayers are always strengthen me. To my wife, Nurul Ain, thank you for your patient and never ending love. And our first daughter, Ummu Haniy, you my dear has been the biggest welcome distraction from my work.

ABSTRACT

Unsaturated polyester resins (UPR) are the most widely used thermosets for composite applications. Besides fiber materials, solid fillers are also used extensively with UPR as reinforcement to enhance thermal and mechanical properties. Recently, the use of high porosity solid such as silica aerogels (SA) in polymer resins is gaining interest. SA is a unique class of nano-porous material with extremely low bulk density and high specific surface area. The addition of SA in polymer resins had resulted in enhanced composite materials having excellent thermal insulation, heat resistance, flame retardancy and lightweight. However, there are two major problems which hindered the production of SA polymer composites on an industrial scale; Firstly, the high cost of conventional SA which depends on expensive chemicals as precursor and secondly, the problem of adsorption of polymer into the SA nano-pores which results to the loss of product properties. As solutions, two approaches were implemented in this study; first was to reduce the production cost via rice husk ash (RHA) as potential silica source for SA synthesis. Second, to prevent resin insertion into SA nano-pores, a novel coating method of the SA particles with polyvinyl alcohol was proposed to provide an impermeable layer to the structure of the SA. Through these approaches, this study aimed to investigate on how the physical, thermal and mechanical behaviours of the UPR composites are affected by changes in certain characteristics of the SA as the filler such as the porosity, particle sizes, surface coating and hybridization. As benchmarking, the amount of filler in UPR was fixed at 30% of volume fraction. The composites were characterized by various methods such as thermogravimetric analysis, differential scanning calorimetry, scanning electron microscopy, uniaxial tensile and compressive tests, dynamic mechanical analysis, hot-disk thermal analyzer and ASTM D635-14 standard for horizontal burning rate. Evaluation of the SA produced from RHA revealed comparable properties to the conventional SA with density of 0.07 g/cm^3, surface area up to 600–700 m^2/g and thermal conductivity as low as 0.04 W/mK. The coating of the SA particles of diameters around 2.5 ± 0.5 mm using a fluidized bed coating technique had resulted in closed–pores core–shell aerogel (CSA) particles with measured shell thickness of between 10-50 µm. For UPR composites filled with silica, the composite containing SA as porous filler was at least 23% and 55% lower in density and thermal conductivity than the composite filled with non-porous filler (precipitated silica) respectively. For the same volume fraction of SA, the improvement in composite's thermal insulation and thermal stability were found to be more for larger SA particles. However, increased in particle size also results in decreased of mechanical properties. For the same particle size, the composite with CSA particles showed a 50% higher of compressive strain and 10 to 12% lower for burning rate as compared to the composite with uncoated SA particles. The CSA particles show reinforcing effects on most of the properties studied, except tensile due to weak filler-matrix bonding. Finally, the combination of SA with alumina trihydrate (ATH) in UPR revealed a synergistic effect during thermal degradation as evidenced by higher thermal stability of the SA/ATH composite when compared to the composite containing only ATH.

ABSTRAK

Resin Poliester tak tepu (UPR) adalah termoset yang paling banyak digunakan bagi aplikasi komposit. Selain bahan gentian, pengisi pepejal juga digunakan secara meluas bersama UPR sebagai penguat bagi menambah baik sifat terma dan mekanikal. Kebelakangan ini, penggunaan pepejal berliang tinggi seperti silika aerogel (SA) dalam resin polimer semakin mendapat perhatian. SA adalah bahan nano berliang yang unik dengan ketumpatan rendah dan luas permukaan spesifik yang tinggi. Penambahan SA dalam resin polimer telah menghasilkan komposit diperhebat dengan penebatan haba, rintangan haba, rencatan api dan ringan. Namun, terdapat dua masalah utama yang menghalang penghasilan SA komposit polimer pada skala industri; pertamanya, kos tinggi bagi SA konvensional yang bergantung pada bahan kimia yang mahal sebagai bahan mula dan kedua, masalah penjerapan polimer ke dalam liang nano SA yang menyebabkan kehilangan sifat produk. Sebagai penyelesaian, dua pendekatan telah dilaksanakan dalam kajian ini; pertama adalah mengurangkan kos pengeluaran melalui abu sekam padi (RHA) sebagai sumber silika yang berpotensi untuk sintesis SA. Kedua, bagi menghalang kemasukkan resin ke dalam liang nano SA, satu kaedah salutan novel bagi partikel SA dengan polivinil alkohol telah dicadangkan bagi menyediakan lapisan tak telap kepada struktur SA itu. Melalui pendekatan tersebut, kajian ini bertujuan untuk menyelidik bagaimana kelakuan fizikal, haba dan mekanikal komposit UPR itu terkesan dengan perubahan ciri-ciri tertentu SA sebagai pengisi seperti keliangan, saiz partikel, salutan permukaan dan penghibridan. Sebagai penanda aras, kandungan pengisi di dalam UPR telah ditetapkan pada 30% pecahan isipadu. Komposit telah dicirikan dengan pelbagai teknik seperti analisis gravimetrik haba, imbasan kebezaan kalorimetri, mikroskop imbasan elektron, ujian ketegangan, mampatan, analisis mekanikal dinamik (DMA), analisis haba cakera - panas dan piawaian ASTM D635-14 untuk kadar pembakaran mendatar. Penilaian SA yang terhasil daripada RHA menunjukkan ciri yang setara dengan SA konvensional dengan ketumpatan 0.07 g/cm^3, luas permukaan sehingga 600-700 m^2/g dan keberaliran haba serendah 0.04 W/mK. Penyalutan partikel SA berdiameter 2.5±0.5 mm menggunakan teknik salutan lapisan terbendalir telah menghasilkan partikel aerogel teras-cengkerang (CSA) yang berliang tutup dengan ketebalan cengkerang 10-50 µm. Untuk komposit UPR yang berisi silika, komposit mengandungi SA sebagai pengisi berliang adalah 23% dan 55% lebih rendah bagi ketumpatan dan keberaliran haba berbanding komposit berisi pengisi tak berliang (silika termendak). Bagi pecahan isipadu SA yang sama, peningkatan penebatan dan kestabilan haba dalam komposit dilihat lebih tinggi bagi partikel SA yang lebih besar. Namun peningkatan saiz partikel juga melemahkan sifat mekanikal. Bagi partikel bersaiz sama, komposit dengan CSA menunjukkan 50% lebih tinggi bagi terikan mampatan dan 10 hingga 12% lebih rendah untuk kadar pembakaran jika dibandingkan dengan komposit berisi partikel SA tak bersalut. Partikel CSA menunjukkan kesan penguatan pada kebanyakkan sifat yang dikaji, kecuali ketegangan kerana ikatan lemah di antara pengisi dan matrik. Akhirnya, gabungan SA dengan alumina trihidrat (ATH) dalam UPR menunjukkan kesan sinergi semasa penguraian haba yang dibuktikan oleh kestabilan haba yang lebih tinggi oleh komposit SA/ATH apabila dibandingkan dengan komposit yang hanya mengandungi ATH.

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LIST OF ABBREVIATIONS

APD	-	Ambient pressure drying
ATH	-	Alumina trihydrate
ATR	-	Attenuated total reflectance
BET	-	Brunauer - Emmett, and Teller
CSA	-	Core – shell aerogel
DMA	-	Dynamic mechanical analysis
DSC	-	Differential scanning calorimetry
DTG	-	Differential thermogravimetric
EDX	-	Energy dispersive X-ray
FBC	-	Fluidized bed coating
FTIR	-	Fourier transform infra red
MEM	-	Modified Elshelby Method
PMC	-	Polymer matrix composite
PSD	-	Particle/pore size distribution
RH	-	Rice husk
RHA	-	Rice husk ash
SA	-	Silica aerogel
SCF	-	Supercritical fluid drying
SEM	-	Scanning electron microscopy
TGA	-	Thermogravimetric analysis
TEOS	-	Tetraethyl orthosilicate
TMOS	-	Tetramethyl orthosilicate
TMCS	-	Trimethyl chloro silane
UPR	-	Unsaturated polyester resin
UPSA	-	Unsaturated polyester / silica aerogel composites
XRD	-	X-ray diffraction

LIST OF SYMBOLS

E'	-	Storage modulus
E''	-	Loss modulus
Tan δ	-	Damping factor
ε	-	Strain
ρ	-	Density
p-SiO_2	-	Precipitated silica
S_v	-	Volumetric shrinkage
W_{abs}	-	Water absorption %
T_g	-	Glass transition temperature
k	-	Thermal conductivity
a	-	Thermal diffusivity
C_p	-	Specific heat capacity
Ø	-	Particle diameter
η_a	-	Adsorption capacity

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Current market segments of polymer matrix composites (PMC) comprise of approximately 80% thermosetting resins or thermosets (Leon, Kim and Helga, 2017, p. 4). Thermosets are widely used for the production of permanent solid structure and dominated the high-temperature applications. Since 1930's, unsaturated polyester resins (UPR) have been intensively used in various sectors covering oil and gas industries, construction, transportation and consumer products owing to its low cost, ease of processing, chemical resistance and strong mechanical properties (Strong, 2008, p. 89). More recently, the development of reinforced UPR composites are gradually replacing steel and concrete in some segments of building and construction industry (Bagherpour, 2012, p. 158; Jabbar and Farid, 2018, p. 216). Due to its low density, a significant reduction in logistic cost can be achieved by using UPR composite materials. Besides, UPR is easier to be molded into intricate design as compared to cast steel, thus considerably reduced the labor cost and fabrication time. UPR market also has increased demands in transportation end-use industry on account of long service life and low maintenance (Gowda, Sanjay and Bhat, 2018, p. 5). Concurrently with this, the needs on improving the performance of UPR have become important in order to meet the requirements of current and future applications.

In general, there are two common approaches to improve the functional properties of polymer matrices. One is intrinsic modification by introducing new reactive species (i.e. monomers) into the polymer chain of the existing resin via co-polymerization process; the other way is additive type modification, which is by physically introduce non-reactive additives or fillers in the polymer resin (Zhang, Huang and Liu, 2011, p. 1768; Lin, Yu and Jin, 2016, p. 633). It is thought that the former technique is more effective in enhancing the resin properties with little detrimental effects on the native properties of the resin. However, this technique showed major drawbacks such as high processing cost and a relatively complicated process. As an alternative, the use of particulate fillers to modify resin properties is often considered as cost-effective due to a variety of low-cost fillers available in the market with high processability. Thus, this area is one of considerable research activity by both industry and academia at the present time.

Regulations on the use of UPR in building and transportation requires deep consideration on human factors, therefore some features such as thermal comfort and fire safety are vital aspects in design (Troitzsc, 2013, p. 2). Currently, conventional UPR based composites often involves high amount of fillers such as sawmill, fumed silica and hollow glass micro balloons to improve thermal insulation, while halogenated compounds and hydrates are the most common material for flame retardancy (Schiavoni, Alessandro, Bianchi and Asdrubali, 2016, p. 990). Moreover, there were also fascinating research works on recycling biomass such as natural fibers as functional fillers in composite materials (Khan, Hameed and Ariffin, 2018, p. 770). Improving the composite performance as well as cost reduction is often thought as values added of using filler. However, cost reduction is not necessarily the case. Although most of the conventional fillers available are cheaper by weight than the resin, PMC fabrication (e.g. molding), however, is more concerned on filler-matrix volume fraction rather than weight (Rothon, 2003, p. 22). Thus, the use of higher density fillers means that volume costs are not reduced by as great an extent.

An effective method in reducing the specific density of a composite material is by the formation of structural pores during composite's fabrication or post-processing. To obtain porosity, the simplest method is by incorporating porous fillers in the polymer matrix itself. Two common characteristics sought after include; lighter weight and control of heat conduction. In this regard, highly porous materials such as silica aerogels (SA) have received an increasing interest in the last decades as filler in polymer composite. Unlike ordinary silica, SA is a special porous material with 3-dimensional mesoporous structure exhibiting many novel properties such as high surface area, ultra low density, excellent thermal insulation and high thermal stability (Soleimani and Abbasi, 2008, p.10). SA is chemically inert and amorphous in nature. It can also be modified into hydrophobic for great dispersion in non-polar resin. Taking into account its unique properties, this study proposes the use of SA as potential filler for UPR with the aim of lightweight-composite materials for high-temperature applications.

1.2 Problems statements

In recent years, there has been growing interest in using SA as filler or reinforcement in polymer resins covering both thermosets and thermoplastics. The SA – polymer composites were characterized by good compressive strength, durability and the most important advantages – lightweight and improved thermal insulation (Salimian, Zadhoush and Naeimirad, 2017, p. 3385). Despite their potential for various applications, the development of the composites however is still far from commercialization due to the expensive cost of SA. Production of conventional SA involves expensive chemicals and energy intensive supercritical drying process. The SA available in the market are derived from silicon alkoxides such as Tetra-ethyl-ortho-silicate (TEOS) and Tetra-methyl-ortho-silicate (TMOS).

Apart from that, there is also a major problem of mixing the SA with liquid resins as intensive immersion of the resin into the porous structure will results in the loss desirable properties. Some studies have demonstrated a direct relationship between preservation of SA pores in the composite and thermal insulation of composites (Chang, Wang and Peng, 2014, p. 8; Kim, Noh and Yu, 2015, p. 40; Liu, Kim and Kwon, 2016, p.1705). To date, a number of methods have been proposed as solutions to prevent the intensive resin intrusion into the SA pores. These methods involve different techniques of polymer-filler mixing and curing but so far, no available study focus on the modification of the SA itself.

Formerly, SA was added in various thermosetting polymers in an attempt to raise the service temperature and insulation performance (Dourbash, Buratti and Belloni, 2017, p. 521; Maghsoudi and Motahari, 2018, p. 706; Guzel, Yilmaz, Deveci, 2018, p. 2; Wang and Jin, 2018, p. 36; Mohamed, Mustaffa and Norizan, 2018, p. 18). However, the use of SA in UPR was only reported by a single group of authors (Mohamed et al, 2013, p. 600; Mohamed et al, 2018, p. 18). Besides, their studies were also limited on thermal stability and flammability of the composites.

In this thesis, economically viable form of SA for composite applications was produced using rice husk. Rice husk is rich silica and its ash can contains more than 85% of active silica by weight. As rice producing country, Malaysia generates more than half millions of tons of rice husks annually, thus can ensure a consistency in raw material. The novelty of this study lies on the approach designed to prevent intensive resin intrusion into the SA structure. Here, the produced SA particles were further coated with polymer into core-shell structure by using fluidized bed coating process. Based on the research gap identified from literatures, this research focuses on investigating the effects of SA on the properties of UPR.

1.3 Research objective and goals

The main objective of this thesis is to prepare and to characterize UPR composite materials using rice husk derived SA as fillers. Therefore this objective will be based on the following strategic goals:

1. To prepare SA particles from rice husk via sol-gel, surface modification and drying process and to characterize the SA particles for comparison with commercial SA.
2. To coat the SA particles with polymer solution into core-shell structured particles by using a bottom spray - fluidized bed coating process.
3. To investigate the effects of SA as fillers with different characteristics such as porosity, particle size, surface coating and hybridization on physical, chemical, mechanical, thermal and flammability properties of the UPR composites.

1.4 Research questions

Pertaining to the strategic goals, the following are the relevant research questions which need to be answered:

1. What are the advantages and disadvantages of rice husk derived SA over chemical derived (commercial) SA? Are their characteristics comparable?
2. What are the added values of modified SA over unmodified/plain SA? Is the proposed coating process feasible?
3. What are the reinforcement and deterioration effects of SA as filler materials on the properties of UPR composites?

1.5 Scope of study

The method of producing sodium silicate solution was based on a patented method (US 7,897,648 B2, Halimaton, 2011). Hydrophilic and hydrophobic SA having particles sizes of not larger than 3.0 mm were produced via supercritical and ambient drying method. Experimental variable in sol-gel process was limited to sol's pH. For a comparison, a high-grade TEOS based SA, was purchased from Cabot Aerogel, United States. Coating of the hydrophobic SA particles was carried out using a lab scale fluidized bed coating machine owned by Universiti Teknikal Melaka. Commercially available, ready to use polyvinyl alcohol (PVA) was used as the coating material and the parameters for coating process were based on optimized process. For polymer matrix, an industrial grade orthophthalic type of UPR was used. Experimental variables for composite blends are limited to the characteristics of the filler, such as porosity, particle size, surface coating and hybridization with filler volume fraction of 30%. As for material characterization, the properties studied are limited to density, morphology, chemical properties, tensile and compressive behaviour, dynamic mechanical properties, thermal degradation and thermal conductivity, curing shrinkage, water absorption and flammability.

REFERENCES

Adeosun, S. O., Gbenebor, O. P., Akpan, E. I., and Udeme, F. A. (2016) 'Influence of Organic Fillers on Physicochemical and Mechanical Properties of Unsaturated Polyester Composites', *Arabian Journal for Science and Engineering*, 41(10), pp. 53–59.

'Aerogel Market Size To Reach $1.92 Billion By 2022. (n.d.)', *Retrieved* January 7, 2017, from https://www.grandviewresearch.com/press-release/global-aerogel-market

'Aerogels Market - Global Industry Analysis, Size, Share, Growth, Trends and Forecast 2015 - 2023. (n.d.)', *Retrieved* January 7, 2017, from http://www.transparencymarketresearch.com/global-aerogels.html

'Aerogel Market is Expected to Reach $1926.6 Million, Globally, by 2021 (n.d.)', Retrieved January 7, 2017, from https://www.alliedmarketresearch.com/aerogel-market

Agrawal, K. C. (2001) *Industrial Power Engineering Handbook.* London: Newnes, pp. 11-14.

Allouche. J., Chaneac. C. and Brayner. R. (2014) 'Design of Magnetic Gelatine/Silica Nanocomposites by Nanoemulsification: Encapsulation versus in Situ Growth of Iron Oxide Colloids', *Nanomaterials (Basel)*, 4(3), pp. 612-637.

Alnaief, M., Antonyuk, S., Hentzschel, C. M., Leopold, C. S., Heinrich, S., and Smirnova, I. (2012) 'A novel process for coating of silica aerogel microspheres for controlled drug release applications', *Microporous and Mesoporous Materials*, 160, pp. 167–173.

Alnaief, M., and Smirnova, I. (2011) 'In situ production of spherical aerogel microparticles', *The Journal of Supercritical Fluids*, 55(3), pp. 1118–1123.

Al-Oweini, R., and El-Rassy, H. (2009) 'Synthesis and characterization by FTIR spectroscopy of silica aerogels prepared using several Si(OR)4 and R″Si(OR′)3 precursors', *Journal of Molecular Structure*, 919(1–3), pp. 140–145.

Anas, S., and Mohamed, R. (2013) 'Thermal Characterisation of Thermoset Polyester Resin Filled Recycle Expanded Polystyrene Composite with Aerogel and Alumina Additives', *Advanced Materials Research*, 664, pp. 600–604

Anderson, A. M., and Carroll, M. K. (2011) 'Hydrophobic Silica Aerogels: Review of Synthesis, Properties and Applications', In *Aerogels Handbook* (pp. 47–77). Springer, New York, NY.

Arroyo, R., Campero, A., & Rodríguez, R. (1993) 'Aggregation profiles of silica sols in a sol-gel process', *Materials Letters*, 16(2), pp. 89–95.

'Asia Pacific Unsaturated Polyester Resin Market is Likely to Reach US$ 7.45 Bn in 2023. (n.d.)', *Retrieved* October 16, 2017, from https://www.transparencymarketresearch.com/pressrelease/asia-pacific-unsaturated-polyester-resin-market.htm

Azlinawati, R., Laftah, W. A., and Shahrir, H. (2013) 'Core–shell polymers: a review', *RSC Advances*, 3(36), pp. 543–565.

Baetens, R., Jelle, B. P., and Gustavsen, A. (2011) 'Aerogel insulation for building applications: A state-of-the-art review', *Energy and Buildings*, 43(4), pp. 761–769.

Bagherpour, S. (2012) *Fibre Reinforced Polyester Composites*. London: InTech, p. 158.

Baker, M., Hollywood, K. A., and Hughes, C. (2016) *Biophotonics: Vibrational Spectroscopic Diagnostics*. San Rafael : Morgan & Claypool Publishers, pp. 1-13.

Bangi, U. K. H., Rao, A. V., and Rao, A. P. (2008) 'A new route for preparation of sodium-silicate-based hydrophobic silica aerogels via ambient-pressure drying'. *Science and Technology of Advanced Materials*, 9(3), pp. 035006.

Bangi, U. K. H., Jung, I.-K., Park, C.-S., Baek, S., and Park, H.-H. (2013) 'Optically transparent silica aerogels based on sodium silicate by a two step sol–gel process and ambient pressure drying'. *Solid State Sciences*, 18, pp. 50–57.

Blout, E. R., and Karplus, R. (1948) 'The Infrared Spectrum of Polyvinyl Alcohol.' *Journal of the American Chemical Society*, 70(2), pp. 862–864.

Budrugeac, P. (2000) 'The evaluation of the non-isothermal kinetic parameters of the thermal and thermo-oxidative degradation of polymers and polymeric materials: its use and abuse', *Polymer Degradation and Stability*, 71(1), pp. 185–187.

Budrugeac, P., and Segal, E. (1997) 'Thermooxidative degradation of an unsaturated polyester resin', *Journal of Thermal Analysis*, 49(1), pp. 183–191.

Chan, L.W., Elaine, S. K. T. and Paul, W. S. H. (2006) 'Comparative study of the fluid dynamics of bottom spray fluid bed coaters', *AAPS PharmSciTech* (7), pp. 45

Chang, S. S. (1992) 'Effect of curing history on ultimate glass transition temperature and network structure of crosslinking polymers', *Polymer*, 33(22), pp. 4768–4778

Chang, K. J., Wang, Y. Z. and Peng, K.. C. (2014) 'Preparation of silica aerogel/polyurethane composites for the application of thermal insulation', *Journal of Polymer Research*, 21(1), pp. 1–9.

Changqing, L., Mao, C. and Dongyi, W. (2017) 'Effect of Filler Shape on the Thermal Conductivity of Thermal Functional Composites', *Journal of Nanomaterials,* 2017, pp. 1-15.

Chaudhuri, R., and Paria, S. (2012) 'Core/Shell Nanoparticles: Classes, Properties, Synthesis Mechanisms, Characterization, and Applications', *Chemical Reviews*, 112(4), pp. 2373–2433.

Chen, H. B., Wang, Y. Z., and Schiraldi, D. A. (2014) 'Preparation and Flammability of Poly(vinyl alcohol) Composite Aerogels', *ACS Applied Materials & Interfaces*, 6(9), pp. 6790–6796.

Coquard, R., Coment, E., Flasquin, G., and Baillis, D. (2013) 'Analysis of the hot-disk technique applied to low-density insulating materials', *International Journal of Thermal Sciences*, 65, pp. 242–253.

Cui, S., Yu, S., Lin, B., Shen, X., and Gu, D. (2015) 'Preparation of SiO 2 aerogel from rice husk ash', *RSC Advances*, 5(81), pp. 65818–65826.

DeArmitt, C. (2011). *26 - Functional Fillers for Plastics,* in M. Kutz (ed.) *Applied Plastics Engineering Handbook*. Oxford: William Andrew Publishing, pp. 455–468.

Della, V. P., Kühn, I., and Hotza, D. (2002) 'Rice husk ash as an alternate source for active silica production', *Materials Letters*, 57(4), pp. 818–821.

Dick, J. S. (1987) 'Compounding materials for the polymer industries: a concise guide to polymers, rubbers, adhesives, and coatings', *Noyes Publications*.

Dodiuk, H., & Goodman, S. H. (2013) *Handbook of Thermoset Plastics*. London: William Andrew, pp. 111-133.

Dourbash, A., Buratti, C., Belloni, E., and Motahari, S. (2017) ‘Preparation and characterization of polyurethane/silica aerogel nanocomposite materials’, *Journal of Applied Polymer Science*, 134(8), pp. 521-530

Du, A., Zhou, B. and Li, Y. (2011) ‘Aerogel: a potential three-dimensional nanoporous filler for resins’, *Journal of Reinforced Plastics and Composites*, 30(11), pp. 912–921.

Eskandari, N., Motahari, S., Atoufi, Z., Hashemi Motlagh, G., and Najafi, M. (2017) ‘Thermal, mechanical, and acoustic properties of silica-aerogel/UPVC composites’, *Journal of Applied Polymer Science*, 134(14).

Fei, S., Wang, L., and Liu, J. (2006) ‘Synthesis and characterization of silica aerogels by a novel fast ambient pressure drying process’, *Materials Letters*, 60(29–30), pp. 3718–3722.

Feng, Q., Chen, K., Ma, D., Lin, H., Liu, Z., Qin, S., and Luo, Y. (2018) ‘Synthesis of high specific surface area silica aerogel from rice husk ash via ambient pressure drying’. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 539, pp. 399–406.

Fruijtier-Pölloth, C. (2012) ‘The toxicological mode of action and the safety of synthetic amorphous silica—A nanostructured material’, *Toxicology*, 294(2–3), pp. 61–79.

Gaishun, V. E., Kosenok, Y. A., Kovalenko, D. L., and Semchenko, A. V. (2008) ‘Sol-Gel Process Preparation of Functional Silica Materials and Their Application’, In *Sol-Gel Methods for Materials Processing* (pp. 297–305). Springer, Dordrecht.

Ge, D., Yang, L., Li, Y., and Zhao, J. (2009) ‘Hydrophobic and thermal insulation properties of silica aerogel/epoxy composite’, *Journal of Non-Crystalline Solids*, 355(52–54), pp. 2610–2615.

‘Global Unsaturated Polyester Resin for Composites Market Top Players Analysis 2018 Dow Chemical, BASF, Polynt. (n.d.)’, *Retrieved* November 11, 2018, from https://thefuturegadgets.com/5486/global-unsaturated-polyester-resin-for-composites-market-top-players-analysis-2018-dow-chemical-basf-polynt/

Goto. K. (1956) ‘Estimation of specific surface area of particles in colloidal silica sols from the rate of dissolution’, *Journal of Physical Chemistry,* 31(8), pp. 900-1007.

Gowda, Y. Sanjay, M.R. and Bhat, K.S. (2018) ‘Polymer matrix-natural fiber composites: An overview’, *Cogent Engineering*, 5(1), pp. 1-13

Guignon, B., Duquenoy, A., and Dumoulin, E. D. (2002) ‘Fluid Bed Encapsulation of Particles: Principles and Practice’, *Drying Technology*, 20(2), pp. 419–447

Guzel Kaya, G., Yilmaz, E., and Deveci, H. (2018) ‘Sustainable nanocomposites of epoxy and silica xerogel synthesized from corn stalk ash: Enhanced thermal and acoustic insulation performance’, *Composites Part B: Engineering*, 150, pp. 1–6.

Gupta, N., and Ricci, W. (2008) ‘Processing and compressive properties of aerogel/epoxy composites’, *Journal of Materials Processing Technology*, 198(1–3), pp. 178–182.

Gurav, J. L., Jung, I. K., Park, H. H. and Kang, E. S. (2010) ‘Silica aerogel: Synthesis and applications’, *Journal of Nanometerials*, pp. 310–323.

Halimaton, H., and Universiti Teknologi Malaysia 2011, *Silica aerogels*, US7897648 B2’, viewed 23 May 2018, retrieved from Google Patents.

Hamouda, A. A., and Amiri, H. A. A. (2014) ‘Factors Affecting Alkaline Sodium Silicate Gelation for In-Depth Reservoir Profile Modification’, *Energies*, 7(2), pp. 568–590.

Hapuarachchi, T. D., Ren, G., Fan, M., Hogg, P. J., and Peijs, T. (2007) ‘Fire Retardancy of Natural Fibre Reinforced Sheet Moulding Compound’, *Applied Composite Materials*, 14(4), pp. 251–264.

Hapuarachchi, T. D., and Peijs, T. (2009) ‘Aluminium trihydroxide in combination with ammonium polyphosphate as flame retardants for unsaturated polyester resin’, *Express Polymer Letters*, 3(11), pp. 743–751.

Heinrich, T., Klett, U., and Fricke, J. (1995) ‘Aerogels—Nanoporous materials part I: Sol-gel process and drying of gels’, *Journal of Porous Materials*, 1(1), pp. 7–17.

Hill, R. R., Muzumdar, S. V., and Lee, L. J. (1995) ‘Analysis of volumetric changes of unsaturated polyester resins during curing’, *Polymer Engineering & Science*, 35(10), pp. 852–859.

Jabbar, S. A., and Farid, S. B. H. (2018) 'Replacement of steel rebars by GFRP rebars in the concrete structures', *Karbala International Journal of Modern Science*, 4(2), pp. 216–227.

Jabbari, M., Åkesson, D., Skrifvars, M., and Taherzadeh, M. J. (2015) 'Novel lightweight and highly thermally insulative silica aerogel-doped poly(vinyl chloride)-coated fabric composite', *Journal of Reinforced Plastics and Composites*, 34(19), pp. 1581–1592.

Jacob, M. (2007) *Chapter 9 Granulation equipment*, in Salman. A. D., Hounslow. M. J. and Seville. J. P. K. (eds.) *Handbook of Powder Technology*. Boston: Elsevier Science B.V., pp. 417–476.

Jung, S. B. and Park, H. H. (2002) 'Control of surface residual -OH polar bonds in SiO2 aerogel film by silylation', *Thin Solid Films*, 420–421, pp. 503–507.

Kabiri, S., Tran, D. N. H., Altalhi, T., and Losic, D. (2014). 'Outstanding adsorption performance of graphene–carbon nanotube aerogels for continuous oil removal'. *Carbon*, 80, pp. 523–533.

Katsumi, G. (1956). 'Effect of pH on Polymerization of Silicic Acid'. *The Journal of Physical Chemistry*, 60(7), pp. 1007–1008.

Khan, T., Hameed Sultan, M. T. B., and Ariffin, A. H. (2018) 'The challenges of natural fiber in manufacturing, material selection, and technology application: A review', *Journal of Reinforced Plastics and Composites*, 37(11), pp. 770–779.

Kim, N. H., Hwang, H. S., Lee, J. Y., Choi. K. H. and Park, I. (2011) Silica Aerogel for the mechanical reinforcement of thermoset epoxy polymers. *18th ICCM*. 21st of August 2011. South Korea, Jeju Island, pp. 1-6.

Kim, G. S., and Hyun, S. H. (2003) 'Effect of mixing on thermal and mechanical properties of aerogel-PVB composites', *Journal of Materials Science*, 38(9), pp. 1961–1966

Kim, C. E., Yoon, J. S., and Hwang, H. J. (2008) 'Synthesis of nanoporous silica aerogel by ambient pressure drying', *Journal of Sol-Gel Science and Technology*, 49(1), pp. 47–52.

Kim, H. M., Kim, H. S., Kim, S. Y., and Youn, J. R. (2015) 'Silica aerogel/epoxy composites with preserved aerogel pores and low thermal conductivity', *E-Polymers*, 15(2), pp. 111–117.

Kim, H. M., Noh, Y. J., Yu, J., Kim, S. Y., and Youn, J. R. (2015) 'Silica aerogel/polyvinyl alcohol (PVA) insulation composites with preserved aerogel pores using interfaces between the superhydrophobic aerogel and hydrophilic PVA solution', *Composites Part A: Applied Science and Manufacturing*, 75, pp. 39–45.

Kim, J., Jeong, D., Son, C., Lee, Y., Kim, E., and Moon, I. (2007) 'Synthesis and applications of unsaturated polyester resins based on PET waste', *Korean Journal of Chemical Engineering*, 24(6), pp. 1076–1083.

Kim, T. R., Joo, K., Lim, B. T., Choi, S. S., Lee, B. J. (2017) 'Enhanced Thermal Conductivity of the Underfill Materials Using Insulated Core/shell Filler Particles for High Performance Flip Chip Applications'. In 2017 *IEEE 67th Electronic Components and Technology Conference (ECTC)* (pp. 1340–1347).

Kim, S. Y., Noh, Y. J. (2014) 'Silica Aerogel/Polyimide Composites with Preserved Aerogel Pores Using Multi-Step Curing', *Macromolecular Research*, 22(1), pp. 108–111.

Kistler, S. S., Fischer, E. A., and Freeman, I. R. (1943) 'Sorption and Surface Area in Silica Aerogel', *Journal of the American Chemical Society*, 65(10), pp. 1909–1919.

Koch, M. L. (1986) *Silica Aerogel and Space Astrophysics*, in Aerogels, *Springer Proceeding in Physics*. Berlin: Springer, pp. 188-192.

Kunii, D., and Levenspiel, O. (2013) *Fluidization Engineering*. Washington: Butterworth-Heinemann, pp. 111–120.

Ladavos A. K. (2012) ' The BET equation, the inflection points of N2 adsorption isotherms and the estimation of specific surface area of porous solids', *Microporous and Mesoporous Materials,* 151, pp. 126 – 133.

Lee, D.-Y., Jin, J. S., and Kang, B. H. (2002). 'Momentum boundary layer and its influence on the convective heat transfer in porous media'. *International Journal of Heat and Mass Transfer*, 45(1), pp. 229–233.

Leon, M., Kim, B., and Helga, P. (2017) 'Materials for Wind Turbine Blades: An Overview', *Materials*, 10(11), pp. 1–4.

Levchik, S. V., and Weil, E. D. (2004) 'A review on thermal decomposition and combustion of thermoplastic polyesters', *Polymers for Advanced Technologies*, 15(12), pp. 691–700.

Levchik, S. V., Levchik, G. F., Camino, G., and Costa, L. (1995) 'Mechanism of Action of Phosphorus-Based Flame Retardants in Nylon 6. II. Ammonium Polyphosphate/Talc', *Journal of Fire Sciences*, 13(1), pp. 43–58.

Levy, F. L. (1981) 'A modified Maxwell-Eucken equation for calculating the thermal conductivity of two-component solutions or mixtures', *International Journal of Refrigeration*, 4(4), pp. 223–225.

Li, Z., Cheng, X., Gong, L., Liu, Q., and Li, S. (2018) 'Enhanced flame retardancy of hydrophobic silica aerogels by using sodium silicate as precursor and phosphoric acid as catalyst', *Journal of Non-Crystalline Solids*, 481, pp. 267–275.

Liauw, C. M., Lees, G. C., Hurst, S. J., Rothon, R. N., and Dobson, D. C. (1996) 'The effect of surface modification of aluminium hydroxide on the crystallisation behaviour of aluminium hydroxide filled polypropylenes'. *Die Angewandte Makromolekulare Chemie*, 235(1), pp. 193–203.

Lin, Y., Yu, B., Jin, X., Song, L., and Hu, Y. (2016) 'Study on thermal degradation and combustion behavior of flame retardant unsaturated polyester resin modified with a reactive phosphorus containing monomer', *RSC Advances*, 6(55), pp. 633–642.

Liu, C., Kim, J. S., and Kwon, Y. (2016) 'Comparative Investigation on Thermal Insulation of Polyurethane Composites Filled with Silica Aerogel and Hollow Silica Microsphere', *Journal of Nanoscience and Nanotechnology*, 16(2), pp. 1703–1707.

Liu, G., Zhou, B., Ni, X., Shen, J., Wu, G., Du, A., and Zu, G. (2012) 'Influence of thermal process on microstructural and physical properties of ambient pressure dried hydrophobic silica aerogel monoliths', *Journal of Sol-Gel Science and Technology*, 62(2), pp. 126–133.

Liu, M., Jiang, W., Chen, Q., Wang, S. (2016) 'A facile one-step method to synthesize SiO2@polydopamine core–shell nanospheres for shear thickening fluid', *RSC Advances*, 6(35), pp. 29279–29287.

Liu, L. X., Litster, J. D., (1996) 'A direct current, plasma fluidized bed reactor: its characteristics and application in diamond synthesis', *Powder Technology,* 88(1), pp. 65–70.

Liu, X., Wang, T., Chow, L. C., Yang, M., and Mitchell, J. W. (2014) ‘Effects of Inorganic Fillers on the Thermal and Mechanical Properties of Poly(lactic acid)’, *International Journal of Polymer Science*, 8(2), pp. 27-28.

Liu, Y. (2016) ‘Frontiers in Aerospace Science: Aerospace Structures and Materials’, *Bentham Science Publishers*.

Lixiao, Z., Wang, Y., Cui, S., Yang, F., Nie, Z., Li, Q., and Wei, Q. (2018) ‘Preparation of Silica Aerogels by Ambient Pressure Drying without Causing Equipment Corrosion’, *Molecules*, 23(8), pp. 1935.

Maghsoudi, K., and Motahari, S. (2018) ‘Mechanical, thermal, and hydrophobic properties of silica aerogel–epoxy composites’, *Journal of Applied Polymer Science*, 135(3), pp. 706-715.

Mallick, P. K. (2017) *Processing of Polymer Matrix Composites: Processing and Applications.* Florida: CRC Press, pp. 5-20.

Mamunya, Y. (2011) *Carbon Nanotubes as Conductive Filler in Segregated Polymer Composites - Electrical Properties*, in Yellampalli. S (ed.) *Carbon Nanotubes - Polymer Nanocomposites*. London: IntechOpen, pp. 8.

Markarian, J. (2003) ‘Flame retardants - higher performance and wider product choice’, *Plastics, Additives and Compounding*, 5(6), pp. 32–36.

Maronga, S. J., Wnukwoski, P. (1997) ‘Modelling of the three-domain fluidized-bed particulate coating process’ 52(17), pp. 219-295.

Martin, J. L. (2007) ‘Kinetic analysis of two DSC peaks in the curing of an unsaturated polyester resin catalyzed with methylethylketone peroxide and cobalt octoate’, *Polymer Engineering & Science*, 47(1), pp. 62–70.

McMorrough, J. (2006) *Materials, Structures, and Standards: All the Details Architects Need to Know But Can Never Find.* Beverly: Rockport Publishers, pp. 229-230.

McNaught, A.D. and Wilkinson, A. (1997) ‘IUPAC Compendium of Chemical Terminology’(2nd ed.)*,* Oxford: Blackwell Science Publications, pp. 112.

Mehrzad, M., Navid, F. M. H., and Mehrdad, K. (2009) ‘Influence of the particle size on the viscoelastic glass transition of silica-filled polystyrene’, *Journal of Applied Polymer Science*, 115(2), pp. 969–975.

Menard, K. P. (2008) *Dynamic Mechanical Analysis: A Practical Introduction*. Boca Raton: CRC Press, pp. 4-232.

Mohamed, R., Mustaffa, S. A. S., Norizan, M. N., and Mohamed, R. R. R. (2018) 'Thermal degradation and flammability of aerogel incorporated UPR/EPS composites with organic nanocrystal presence', *AIP Conference Proceedings*, 1985(1), pp. 18-22.

Motahari, S., Motlagh, G. H., and Moharramzadeh, A. (2015) 'Thermal and Flammability Properties of Polypropylene/Silica Aerogel Composites', *Journal of Macromolecular Science, Part B*, 54(9), pp. 1081–1091.

Murafa, A. V., Bobyreva, N. I., and Khozin, V. G. (1996) 'Modification of polyester resins with active mineral fillers', *Mech Compos Mater*, 32(1), pp. 82-86.

National Center for Biotechnology Information. PubChem Database. Tetraethyl orthosilicate, https://pubchem.ncbi.nlm.nih.gov/compound/Tetraethyl-orthosilicate, CID 6517. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Tetrameth oxysilane, https://pubchem.ncbi.nlm.nih.gov/compound/Tetramethoxysilane, CID 12682. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Sodium silicate, https://pubchem.ncbi.nlm.nih.gov/compound/Sodium-silicate, CID 23266. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Ethanol, https://pubchem.ncbi.nlm.nih.gov/compound/Ethanol, CID=702 (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Methanol, https://pubchem.ncbi.nlm.nih.gov/compound/Methanol, CID=887. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Carbon dioxide, https://pubchem.ncbi.nlm.nih.gov/compound/Carbon-dioxide, CID=280. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Acetone, https://pubchem.ncbi.nlm.nih.gov/compound/Acetone, CID=180. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Toluene, https://pubchem.ncbi.nlm.nih.gov/compound/Toluene, CID=1140. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Heptane, https://pubchem.ncbi.nlm.nih.gov/compound/Heptane, CID=8900. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Hexane, https://pubchem.ncbi.nlm.nih.gov/compound/Hexane, CID=8058. (accessed on Aug. 20, 2018)

National Center for Biotechnology Information. PubChem Database. Pentane, https://pubchem.ncbi.nlm.nih.gov/compound/Pentane, CID=8003. (accessed on Aug. 20, 2018)

Nayak, J. P., and Bera, J. (2009) 'Preparation of Silica Aerogel by Ambient Pressure Drying Process using Rice Husk Ash as Raw Material', *Transactions of the Indian Ceramic Society*, 68(2), pp. 91–94.

Nelson, A., Wang, D., Koynov, K., and Isa, L. (2015) 'A multiscale approach to the adsorption of core–shell nanoparticles at fluid interfaces', *Soft Matter*, 11(1), pp. 118–129.

Nienow, A. W. and Chiba. T (1985) *Studies of segregation mixing in fludized beds of different size particles,* in Davidson. J., F., Clift. R and Harrison. D. (eds.), *Fluidization*. London: Academic Press, pp. 357-392.

Nirmala, G., Loganathan, M., and Kumar, P. (2014) 'Viscosity Effects on Solid Circulation Rate in a Liquid Solid Circulating Fluidized Bed', *Journal of Applied Sciences*, 14, pp. 1037-1042.

Oda, Y., and Shinke, Y. (2014) *Vinyl Polymers,* In Kobayashi, S and Müllen, L (eds.), *Encyclopedia of Polymeric Nanomaterials*. Heidelberg: Springer Berlin, pp. 160-170.

Omamogho, J. O., Hanrahan, J. P., Tobin, J., and Glennon, J. D. (2011) 'Structural variation of solid core and thickness of porous shell of 1.7μm core–shell silica particles on chromatographic performance: Narrow bore columns', *Journal of Chromatography A*, 1218(15), pp. 1942–1953.

Panasyuk, G. P., Belan, V. N., Voroshilov, I. L., Kozerozhets, I. V., Luchkov, I. V., Kondakov, D. F., and Demina, L. I. (2013) 'The study of hydrargillite and γ-alumina conversion process in boehmite in different hydrothermal media', *Theoretical Foundations of Chemical Engineering*, 47(4), pp. 415–421.

Park, Y. K., Lee, J.-K., and Kim, J.-G. (2008) ‘A New Approach to Predict the Thermal Conductivity of Composites with Coated Spherical Fillers and Imperfect Interface’, *Materials Transactions*, 49(4), pp. 733–736.

Peng, Z., and Kong, L. X. (2007) ‘A thermal degradation mechanism of polyvinyl alcohol/silica nanocomposites’, *Polymer Degradation and Stability*, 92(6), pp. 1061–1071.

Perrut, M. (2000) ‘Supercritical Fluid Applications: Industrial Developments and Economic Issues’, *Industrial & Engineering Chemistry Research*, 39(12), pp. 4531–4535.

Peters, S. T. (2013) *Handbook of Composites*. Berlin: Springer Science & Business Media, pp. 249-250

Pisal, A. A., and Rao, A. V. (2016) ‘Comparative studies on the physical properties of TEOS, TMOS and Na2SiO3 based silica aerogels by ambient pressure drying method’, *Journal of Porous Materials*, 23(6), pp. 1547–1556.

Pradip B. Sarawade, J.-K. K., and Jin-Koo Park, H.-K. K. (2006) ‘Influence of Solvent Exchange on the Physical Properties of Sodium Silicate Based Aerogel Prepared at Ambient Pressure’, *Aerosol and Air Quality Research*, 6(1), pp. 93–105.

Prakash, S. S., Brinker, C. J., Hurd, A. J., and Rao, S. M. (1995) ‘Silica aerogel films prepared at ambient pressure by using surface derivatization to induce reversible drying shrinkage’, *Nature*, 374(6521), pp. 439–443.

Qe, H., Huang, D., and He, S. (2013) ‘Thermal Protective Performance of Aerogel Embedded Firefighter’s Protective Clothing’, *Journal of Engineered Fibers and Fabrics*, 8(2), pp. 134–140.

Qu, L., Choy, K.-L., and Wheatley, R. (2016) ‘An Atomistic-Scale Study for Thermal Conductivity and Thermochemical Compatibility in (DyY)Zr2O7 Combining an Experimental Approach with Theoretical Calculation’, *Scientific Reports*, 6, pp. 232.

Rafiee, E., Shahebrahimi, S., Feyzi, M., and Shaterzadeh, M. (2012) ‘Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material’, *International Nano Letters*, 2(1), pp. 2-9.

Raj, B., Voorde, M. V. de, and Mahajan, Y. (2017) *Nanotechnology for Energy Sustainability*. Weinheim: John Wiley & Sons, pp. 944.

Rao, A. P., & Rao, A. V. (2009) ‘Modifying the surface energy and hydrophobicity of the low-density silica aerogels through the use of combinations of surface-modification agents’, *Journal of Materials Science*, 45(1), pp. 51–63.

Rassy, H., and Pierre, A. C. (2005) ‘NMR and IR spectroscopy of silica aerogels with different hydrophobic characteristics’, *Journal of Non-Crystalline Solids*, 351(19–20), pp. 1603–1610.

Rechberger, F., and Niederberger, M. (2017) ‘Synthesis of aerogels: from molecular routes to 3-dimensional nanoparticle assembly’, *Nanoscale Horizons*, 2(1), pp. 6–30.

Reynolds, J. G., Coronado, P. R., and Hrubesh, L. W. (2001). ‘Hydrophobic aerogels for oil-spill clean up – synthesis and characterization’. *Journal of Non-Crystalline Solids*, 292(1), pp. 127–137.

Richard, G., Edward, S., Metanomski, W. V., and Kahovec, J. (2009) ‘Compendium of Polymer Terminology and Nomenclature: IUPAC Recommendations 2008’, *Royal Society of Chemistry*.

Rothon, R. (2003) ‘Particulate-filled Polymer Composites’. *iSmithers Rapra Publishing*, pp. 20–180.

Rouquerol, J., Rouquerol, F., and Ganteaume, M. (1975) ‘Thermal decomposition of gibbsite under low pressures: I. Formation of the boehmitic phase’, *Journal of Catalysis*, 36(1), pp. 99–110.

Rozman, H. D., Musa, L., & Abubakar, A. (2005) ‘Rice husk–polyester composites: The effect of chemical modification of rice husk on the mechanical and dimensional stability properties’, *Journal of Applied Polymer Science*, 97(3), pp. 1237–1247.

Rybak, A., and Gaska, K. (2015) ‘Functional composites with core–shell fillers: I. Particle synthesis and thermal conductivity measurements’, *Journal of Materials Science*, 50(23), pp. 7779–7789.

Rybak, A., Gaska, K., Kapusta, C., Toche, F., and Salles, V. (2017) ‘Epoxy composites with ceramic core–shell fillers for thermal management in electrical devices’, *Polymers for Advanced Technologies*.

Sahari, J., and Maleque, M. A. (2016) ‘Effect of oil palm ash on the mechanical and thermal properties of unsaturated polyester composites’, *E-Polymers*, 16(4), pp. 323–329.

Salamone, J. C. (1998) *Concise Polymeric Materials Encyclopedia.* Boca Raton: CRC Press, pp. 1590-1599.

Salimian, S., Zadhoush, A., Naeimirad, M., Kotek, R., and Ramakrishna, S. (2018) 'A review on aerogel: 3D nanoporous structured fillers in polymer-based nanocomposites', *Polymer Composites*, 39(10), pp. 3383–3408.

Salla, J. M., & Martín, J. L. (1988) 'Dynamic, isothermal and residual heats of curing of an unsaturated polyester resin', *Thermochimica Acta*, 126, pp. 339–354.

Salman, Z. (2011) *Renewable energy in Southeast Asia: Agricultural biomass waste*. Routledge: Chapman & Hall.

Samanta, S., Pescatore, P. F., Thomas, B. P. and Cabot Corp, *'Aerogel blanket and method of production'* US20140273701, viewed 12 July 2018, retrieved from Google Patents.

Retrieved from http://www.google.com/patents/US20140273701

Sasihithlu, K., Pendry, J. B., and Craster, R. V. (2016) 'Van der Waals force assisted heat transfer for vacuum gap spacings', *Z. Naturforsch*, 72(2), pp. 181-188.

Sato, T. (1959) 'The dehydration of alumina trihydrate', *Journal of Applied Chemistry*, 9(6), pp. 331–340.

Scheirs, J., and Long, T. E. (2005) 'Modern Polyesters: Chemistry and Technology of Polyesters and Copolyesters', *John Wiley & Sons*.

Schiavoni, S., D'Alessandro, F., Bianchi, F., and Asdrubali, F. (2016) 'Insulation materials for the building sector: A review and comparative analysis', *Renewable and Sustainable Energy Reviews*, 62, pp. 988–1011.

Schwertfeger, F., Glaubitt, W., and Schubert, U. (1992) 'Hydrophobic aerogels from Si(OMe)4/MeSi(OMe)3 mixtures', *Journal of Non-Crystalline Solids*, 145, pp. 85–89.

Şenol, D., and Kaya, İ. (2017) 'Synthesis and characterization of azomethine polymers containing ether and ester groups', *Journal of Saudi Chemical Society*, 21(5), pp. 505–516

Shaid, M. F. (2014) 'Thermophysiological Comfort Analysis of Aerogel Nanoparticle Incorporated Fabric for Fire Fighter's Protective Clothing', *Chemical and Materials Engineering*, 2(2), pp. 37–43.

Shashikanta, S. A., Patil, R., and Ekbote, T. (2015) 'Investigation of Mechanical and Thermal Behaviour of Aluminium Hydroxide/Epoxy composite filled with

Silica Aerogel Material', *International Journal of Mechanical Engineering*, 2(11), pp. 1–5.

Shewale, P. M., Rao, A. V., and Rao, A. P. (2008). 'Effect of different trimethyl silylating agents on the hydrophobic and physical properties of silica aerogels', *Applied Surface Science*, 254(21), pp. 6902–6907.

Shonaike, G. O., and Simon, G. P. (1999) *Polymer Blends and Alloys*. Berlin: CRC Press, pp. 192-193

Sing, K. S. W., Everett. D. H. and Haul. R. A. W. (1985) 'Reporting Physiosorption Data for Gas/Solid Systems with Special Referenceto the Determination of Surface Area and Porosity', *Pure Applied Chemistry,* 57, pp. 603

SK S. Hossain, Lakshya Mathur and P.K. Roy (2018) 'Rice husk/rice husk ash as an alternative source of silica in ceramics: A review', *Journal of Asian Ceramic Societies*, 6(4), pp. 299-313.

Soares, C. A. M., Soares, C. M. M., and Freitas, M. J. M. (1999) *Mechanics of Composite Materials and Structures*. Berlin: Springer Science & Business Media, pp. 285-290.

Soleimani Dorcheh, A., and Abbasi, M. H. (2008) 'Silica aerogel; synthesis, properties and characterization', *Journal of Materials Processing Technology*, 199(1–3), pp. 10–26.

Štandeker, S., Novak, Z., and Knez, Ž. (2007) 'Adsorption of toxic organic compounds from water with hydrophobic silica aerogels', *Journal of Colloid and Interface Science*, 310(2), pp. 362–368.

Strong, A. B. (2008) 'Fundamentals of Composites Manufacturing' (2^{nd} ed.) *Materials, Methods and Applications*. Michigan: Society of Manufacturing Engineers, pp. 86-89.

Tadjarodi, A., Haghverdi, M., and Mohammadi, V. (2012) 'Preparation and characterization of nano-porous silica aerogel from rice husk ash by drying at atmospheric pressure', *Materials Research Bulletin*, 47(9), pp. 2584–2589.

Tajiri, K., Igarashi, K., and Nishio, T. (1995) 'Effects of supercritical drying media on structure and properties of silica aerogel', *Journal of Non-Crystalline Solids*, 186, pp. 83–87.

Tang, G. H., Bi, C., and Fu, B. (2013) 'Thermal conduction in nano-porous silicon thin film', *Journal of Applied Physics*, 114(18), pp. 184.

Tawfik, S. Y., Asaad, J. N., and Sabaa, M. W. (2004) 'Studies on Polymeric Composites Containing Alumina Trihydrate and Aswan Clay Fillers', *Polymer-Plastics Technology and Engineering*, 43(1), pp. 57–79.

Temel, T. M., İkizler, B. K., Terzioğlu, P., Yücel, S., and Elalmış, Y. B. (2017) 'The effect of process variables on the properties of nanoporous silica aerogels: an approach to prepare silica aerogels from biosilica', *Journal of Sol-Gel Science and Technology*, 84(1), pp. 51–59.

Thommes. M. (2015) 'Physisorption of gases, with special reference to the evaluation of surface area and pore size distribution (IUPAC technical report)', *Pure Applied Chemistry,* pp. 1 – 19.

Ting, L., & Wang, T. (2008) 'Preparation of silica aerogel from rice hull ash by drying at atmospheric pressure', *Materials Chemistry and Physics*, 112(2), pp. 398–401.

Troitzsc, J. H. (2013) 'Fires, Fire Safety and Trends. Regulations, Standards and the Role of Flame Retardancy', *International Polymer Science and Technology*, 40(10), 1–6.

Ülkü, S., Balköse, D., & Baltacboğlu, H. (1993) 'Effect of preparation pH on pore structure of silica gels', *Colloid and Polymer Science*, 271(7), pp. 709–713.

'Unsaturated Polyester Resins Market is forecast to expand at a CAGR of 5.0% by 2022. (n.d.)', *Retrieved* January 18, 2018, from https://www.transparencymarketresearch.com/pressrelease/unsaturated-polyester-resin.htm

Venkateswara Rao, A., and Kalesh, R. R. (2003) 'Comparative studies of the physical and hydrophobic properties of TEOS based silica aerogels using different co-precursors', *Science and Technology of Advanced Materials*, 4(6), pp. 509–515.

Vilay, V., Mariatti, M., Mat Taib, R., and Todo, M. (2008) 'Effect of fiber surface treatment and fiber loading on the properties of bagasse fiber–reinforced unsaturated polyester composites', *Composites Science and Technology*, 68(3), pp. 631–638.

Walczak, E. (1999). 'New ecological polyester resins with reduced flammability and smoke evolution capacity', *Polymer Degradation and Stability*, 64(3), pp. 439–442.

Walczak, E. (2003) 'Kinetics of thermal decomposition of unsaturated polyester resins with reduced flammability', *Journal of Applied Polymer Science*, 88(13), pp. 2851–2857.

Wang, J. (2018) 'Preparation and properties of SiO2 aerogel and fabric composite based on polyurethane', *Integrated Ferroelectrics*, 189(1), pp. 36–43.

Wang, Y., Zhang, L., Yang, Y., and Cai, X. (2016) 'Synergistic flame retardant effects and mechanisms of aluminum diethylphosphinate (AlPi) in combination with aluminum trihydrate (ATH) in UPR', *Journal of Thermal Analysis and Calorimetry*, 125(2), pp. 839–848.

Weatherhead, R. G. (1980) *Catalysts, Accelerators and Inhibitors for Unsaturated Polyester Resins,* In *FRP Technology*. Dordrecht: Springer, pp. 204–239.

Wetton, R. E., Marsh, R. D. L., and Van-de-Velde, J. G. (1991) 'Theory and application of dynamic mechanical thermal analysis', *Thermochimica Acta*, 175(1), pp. 1–11.

Williams, M. K.. and Robertson, L. B. (2011) Flame Retardant Effects Of Aerogel And Nanosilica On Engineered Polymers. ICCE-18 International Community of Composite Engineering. 4th July 2010. Shanghai, China, pp. 1-6.

Worzakowska, M. (2006) 'Kinetics of the curing reaction of unsaturated polyester resins catalyzed with new initiators and a promoter', *Journal of Applied Polymer Science*, 102(2), pp. 1870–1876.

Xu, G. F. H., and Yuqiang Guo, J. T. (2013) 'Preparation and Characterization of Hydrophobic Silica Aerogel by Ambient Pressure Drying Method', *Key Engineering Materials*, 544, pp. 144–147.

Xu, J. Z., Gao, B. Z., and Kang, F. Y. (2016) 'A reconstruction of Maxwell model for effective thermal conductivity of composite materials', *Applied Thermal Engineering*, 102, pp. 972–979.

Xu, L., & Lee, L. J. (2005) 'Kinetic analysis and mechanical properties of nanoclay reinforced unsaturated polyester (UP) resins cured at low temperatures', *Polymer Engineering & Science*, 45(4), pp. 496–509.

Xu, Z., Yan, L., & Chen, L. (2016) 'Synergistic Flame Retardant Effects between Aluminum Hydroxide and Halogen-free Flame Retardants in High Density Polyethylene Composites', *Procedia Engineering*, 135, pp. 631–636.

Yasushi, S., and Norihiko, K. (2004) ‘Quantitative Analysis of Tridymite and Cristobalite Crystallized in Rice Husk Ash by Heating’, *Industrial Health*, 42(2), pp. 277–285.

Zhang, C., Huang, J. Y., Liu, S. M., and Zhao, J. Q. (2011) ‘The synthesis and properties of a reactive flame-retardant unsaturated polyester resin from a phosphorus-containing diacid’, *Polymers for Advanced Technologies*, 22(12), pp. 1768–1777.

Zhang, X., Heinonen, S., and Levänen, E. (2014) ‘Applications of supercritical carbon dioxide in materials processing and synthesis’, *RSC Advances*, 4(105), pp. 137–152.

Zhao, J. P., Ge, D. T., Zhang, S. L., and Wei, X. L. (2007) ‘Studies on Thermal Property of Silica Aerogel/Epoxy Composite’, *Materials Science Forum*, 546–549, pp. 1581–1584.

Zhihua, Z., Jun, S., Xingyuan, N., Yang, L. (2008) ‘Benzene adsorption properties of silica aerogel-fiber composites’. In *2008 2nd IEEE International Nanoelectronics Conference* (pp. 366–370).

Zhou, X., Zhong, L., and Xu, Y. (2008) ‘Surface modification of silica aerogels with trimethylchlorosilane in the ambient pressure drying’, *Inorganic Materials*, 44(9), pp. 976–979.

Zhu, B., Fang, B., and Li, X. (2010) ‘Dehydration reactions and kinetic parameters of gibbsite’, *Ceramics International*, 36(8), pp. 2493–2498.

Zhu, Y., Otsubo, M., Honda, C., and Ohno, A. (2005) ‘Suppression effect of ATH filler on the erosion of filled silicone rubber exposed to dry band arc discharge’, *Polymer Testing*, 24(7), pp. 893–899.

Zuo, L., Zhang, Y., Zhang, L., Miao, Y.-E., Fan, W., & Liu, T. (2015). ‘Polymer/Carbon-Based Hybrid Aerogels: Preparation, Properties and Applications’, *Materials*, 8(10), pp. 6806–6848.

LIST OF PUBLICATIONS

Journal with Impact Factor

1. Zulhelmi Alif Abdul Halim, Muhamad Azizi Mat Yajid, Mohd Hasbullah Idris, and Halimaton Hamdan. 2017. "Effects of Silica Aerogel Particle Sizes on the Thermal–Mechanical Properties of Silica Aerogel – Unsaturated Polyester Composites." Plastics, Rubber and Composites 46 (4): 184–92. https://doi.org/10.1080/14658011.2017.1306913. **(Q3, IF: 0.848)**

2. Zulhelmi Alif Abdul Halim, M Azizi Mat Yajid, M Hasbullah Idris, and H Hamdan. 2018. "Dispersion of Polymeric-Coated–Silica Aerogel Particles in Unsaturated Polyester Composites: Effects on Thermal-Mechanical Properties." Journal of Dispersion Science and Technology 39 (8): 1093–1101. https://doi.org/10.1080/01932691.2017.1382375. **(Q2, IF: 1.454)**

3. Zulhelmi Alif Abdul Halim, Muhamad Azizi Mat Yajid, Mohd Hasbullah Idris, and Halimaton Hamdan. 2018. "Physiochemical and Thermal Properties of Silica Aerogel–Poly Vinyl Alcohol / Core–Shell Structure Prepared Using Fluidized Bed Coating Process for Thermal Insulation Applications." Materials Chemistry and Physics 215 (August): 269–76. https://doi.org/10.1016/j.matchemphys.2018.05.019. **(Q2, IF: 2.210)**

4. Zulhelmi Alif Abdul Halim, Muhamad Azizi M. Yajid, M. Hasbullah Idris, and Halimaton Hamdan. 2018. "Effects of Rice Husk Derived Amorphous Silica on the Thermal-Mechanical Properties of Unsaturated Polyester Composites." Journal of Macromolecular Science, Part B 57 (6): 479–96. https://doi.org/10.1080/00222348.2018.1476440. **(Q3, IF: 1.005)**

5. Zulhelmi Alif Abdul Halim, Muhamad Azizi M. Yajid and Halimaton Hamdan, 2020. Effects of Solvent Exchange Period and Heat Treatment on Physical and Chemical Properties of Rice Husk Derived Silica Aerogels. Silicon (2020). https://doi.org/10.1007/s12633-020-00421-5 **(Q4, IF: 1.202)**

Indexed Journal

1. Zulhelmi Alif Abdul Halim, Muhamad Azizi Mat Yajid, and Halimaton Hamdan. 2016. "Synthesis and Characterization of Rice Husk Ash Derived - Silica Aerogel Beads Prepared by Ambient Pressure Drying." Key Engineering Materials 694: 106–10. https://doi.org/10.4028/www.scientific.net/KEM.694.106. **(Indexed by SCOPUS)**

2. Zulhelmi Alif Abdul Halim, Muhamad Azizi Mat Yajid, and Halimaton Hamdan. 2017. "Thermal Gravimetric Analysis of Unsaturated Polyesters Filled with Alumina Trihydrate and Silica Aerogel". Solid State Phenomena 264: 116–119. https://doi.org/10.4028/www.scientific.net/SSP.264.116 **(Indexed by SCOPUS)**

3. Zulhelmi Alif Abdul Halim, Yajid, M. A. M., & Idris, M. H. (2019). Effect of wash solvents on salt removal in rice husk derived nano-silica. AIP Conference Proceedings, 2068(1), 020106. https://doi.org/10.1063/1.5089405 **(Indexed by SCOPUS)**

4. Z.A.A. Halim, M.A.M. Yajid, (2019). Comparison of Dynamic Mechanical Properties and Thermal Conductivity of Unsaturated Polyester Composites Filled With Plain SiO2 Aerogel and Core-Shell SiO2 Aerogel, Materials Today: Proceedings 17:3,686-693. https://doi.org/10.1016/j.matpr.2019.06.351. **(Indexed by SCOPUS)**

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