# Mechanical Properties and Moisture Absorption of Epoxy Composites Mixed with Amorphous and Crystalline Silica from Rice Husk

Norul Hisham Hamid,<sup>a,b</sup> Wan Sulwani Izzati Wan Baderul Hisan,<sup>a</sup> Ummi Hani Abdullah,<sup>b</sup> Ammir Affan Abdul Azim,<sup>b</sup> and Paridah Md. Tahir <sup>a</sup>

Thermosetting epoxies are low-density polymers with excellent adhesion, mechanical properties, and resistance to environmental degradation. They are widely used as molds and castings for manufacturing, protective coatings, and reinforcement material for aircraft. However, polymer brittleness is a major disadvantage preventing its widespread application. The addition of filler materials such as nanoparticles has attracted considerable attention. Using reinforcement materials for biocomposite derived from a natural, renewable and sustainable material will lower the manufacturing cost for epoxy composites. This study investigated the physical and mechanical properties of epoxy composites reinforced with amorphous and crystalline silica. The amorphous and crystalline silica were obtained by the precipitation method from the carbonization of rice husk at 700 °C and 1000 °C for 6 h, separately. The epoxy resin was mixed with 5 wt%, 10 wt%, and 15 wt% concentrations (by weight) of amorphous and crystalline silica, separately. Silica 10 nm to 30 nm in size was obtained, as observed by scanning electron microscopy (SEM). The epoxy nano-crystalline silica composite had a significantly lower thickness swelling value compared to the epoxy nano-amorphous silica composite. However, the epoxy nano-crystalline silica composite had significantly better mechanical properties.

#### Keywords: Composite; Epoxy; Nano-silica; Rice husk

Contact information: a: Biocomposite Technology and Design Unit, Institute of Tropical Forestry & Forest Products, Universiti Putra Malaysia, 43400, UPM, Serdang, Selangor Darul Ehsan, Malaysia; b: Department of Forest Production, Faculty of Forestry, Universiti Putra Malaysia, 43400, UPM, Serdang, Selangor Darul Ehsan, Malaysia; \*Corresponding author: h\_noroul@upm.edu.my

### INTRODUCTION

The use of natural fibers in polymer composites has attracted much attention from researchers due to their renewable and sustainable properties. Natural fiber polymer composites (NFPCs) have varying properties depending on the fiber type, the fiber source, and the fiber structure. In many cases, biocomposites made from natural fibers reinforced with thermoplastic or thermosetting polymers have excellent mechanical properties in terms of their flexural strength, flexural modulus, fracture toughness, and impact strength (Bénézet *et al.* 2012; Shalwan and Yousif 2013). The improvement of biocomposite strength properties is attributed to the ability of cellulose fibers to withstand bending force and resist fracture force. The influences of different chemical treatments and modifications on the properties of natural fibers combined with thermosetting or thermoplastic polymers have been well researched (Ticoalu *et al.* 2010; Faruk *et al.* 2012; Arrakhiz *et al.* 2013, Di Bella *et al.* 2014). The modification of banana fibers with 1wt% sodium hydroxide (NaOH) created composites with improved mechanical properties (Venkateshwaran *et al.* 2013).

The use of NFPCs is limited due to their high water absorption and inferior fire resistance. Oil palm lignin reinforced with epoxy suffers from high water absorption and dimensional instability when exposed to prolonged high humidity conditions (Abdul Khalil *et al.* 2011).

Natural fibers are abundant with cellulose, hemicellulose, lignin, and pectin. These structural chemical components contain hydroxyl 1 groups, which are hydrophilic, unlike the hydrophobic polymers. This presents a challenge to match the most suitable matrix and fiber to overcome the weaker interfacial area between the matrices and the fibers. The weaker interface will absorb more water from the composite surface and the absorption rate will gradually decrease towards the center (Xie *et al.* 2009). The high water uptake increases the composite weight, reduces its strength, increases the deflection, increases the swelling (causing warping), increases buckling, and promotes microbial degradation (Ab Ghani and Ahmad 2011).

A similar trend was observed in oil palm/natural rubber composites. The increased water uptake was due to the higher hydrophobicity of the fibers. The addition of fibers caused the water absorption properties of natural rubber to change from Fickian law to non-Fickian. This happened due to the microcracks and viscoelasticity nature of the polymer (Kabir *et al.* 2012). In the woven pandanus and banana fabric composite tests, the woven pandanus fabric composites had a higher water uptake compared to the woven banana fabric composites because of the higher lignin and hemicellulose content, as well as the presence of defects in the composite. Furthermore, the temperature can affect the water absorption of the composites (Arrakhiz *et al.* 2013).

The water absorption properties of oil palm fiber-natural rubber (OPF-NR) was less than that of the OPF-sisal fiber-NR hybrid biocomposite. Sisal fiber contains a relatively high amount of holocellulose (23%), which is exceptionally hydrophilic and therefore led to a higher water intake. Additionally, the lignin content in OPF (19%) was higher than that of sisal fiber (9%). Since lignin is hydrophobic, its presence reduces the water absorption (Shinoj *et al.* 2011).

Many studies reported the reduction of moisture adsorption in NFPCs by applying coupling agents such as maleic anhydride and polyethylene, as well as the modification of fibers by bleaching, acetylation, and alkaline treatment (Shinoj *et al.* 2011). During the chemical treatment, the fibers are cleaned to ensure their purity, and the moisture absorption is eliminated by discharging the OH groups in the fibers (Kabir *et al.* 2012; Shalwan and Yousif 2013).

Strong moisture absorption properties may limit the application of NFPCs as aerospace and automotive composites for interior application, especially when they are used in environments with fluctuating humidity. Many natural fibers are modified with silane treatment, gamma irradiation, latex coating, mercerization, acetylation, and peroxide to increase their hydrophobicity. These modification treatments require more money, extra processing equipment, and more time. Exploring other hydrophobic materials that can be integrated with polymers is critically needed. This study used nano-amorphous and nanocrystalline silicas from rice husk to improve the hydrophobic properties of epoxy biocomposites.

According to the Malaysian Ministry of Agriculture; about 408,000 metric tonnes of rice husk are produced in Malaysia annually (Rashid *et al.* 2010). Considering the 29 to 38% of cellulose, 9 to 20% of lignin, and 18.8 to 22.3% of silica oxide in rice husk as reported by Jauberthie *et al.* (2000); this will generate the potential of 784,000 to 1,064,000 tonne of cellulose, 252,000 to 560,000 tonne of lignin, and 526,400 to 624,400 tonne of silica oxide. However, currently rice husk is used as fuel to produce hot air for drying the

rice (Idros 2012). In 2009, many reports and complaints were issued regarding the health impact of rice husk dust towards the residents. Generally, farmers and rice processor often burn the rice husk openly as wastes, and this release carbon dioxide ( $CO_2$ ) into the atmosphere.  $CO_2$  is a well-known greenhouse gas (Rashid *et al.* 2010; Shuhadah and Rohasliney 2011). These pollutants have been linked to several health problems, including asthma, respiratory illnesses, nervous system damage, kidney and liver damage, and reproductive or developmental disorders. Therefore, it is vital to extract the structural organic chemistry from rice husk to manufacture and use for other usable products.

The novelty of study is using the amorphous and crystalline silica from Malaysia's rice husk. It may be different from other countries rice husk such as European or Thailand. This may due to different weather, soil, climate, and agriculture practices (Lim *et al.* 2012). The other novelty is that this study used natural silica from natural fibre. This natural silica is totally different from other commercial silica products. The silica from rice husk is more sustainable and environmental friendly (Sae-Oui *et al.* 2002).

# EXPERIMENTAL

### Materials

The rice husk and epoxy resin used in this study were obtained from Bintang Kencana Fibrecomposite Sdn. Bhd (Kuala Lumpur, Malaysia) and Asachem (M) Sdn. Bhd (Selangor, Malaysia). All chemicals, which were hydrochloric acid (HCl), sodium hydroxide (NaOH), and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), were supplied by Merck (M) Sdn. Bhd (Petaling Jaya, Malaysia).

# Methods

### Preparation of nano-silica

The nano-silica was prepared according to the method adopted by Monshizadeh et al. (2012). The rice husk ash was washed with distilled water and burned separately at 700 °C (nano-amorphous silica) and 1000 °C (nano-crystalline silica) for 6 h (Hamad and Khattab 1981; Patel et al. 1987; Sidheswaran and Bhat 1996; Yalcin and Sevinc 2001). Ten grams of rice husk ash (RHA) samples were stirred in 80 mL of 2.5 N NaOH solution. The RHA solution was then boiled in a covered 250 mL Erlenmeyer flask for 3 h. The solution was filtered, and the residue was washed with 20 mL of boiling water. The filtrate cooled to room temperature and 5 N sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) was added until a pH of 2 was reached, after which ammonium hydroxide (NH<sub>4</sub>OH) was added until a pH 8.5 was reached. The solution was maintained at room temperature for 3.5 h. The filtrate was then dried in an oven (DHG 9070A Dry Oven, Zenith Lab Inc, China) at 120 °C for 12 h. Pure silica was extracted by refluxing with 6 N hydrochloric acid (HCl) for 4 h. The pure silica was then was washed repeatedly using deionized water to achieve a neutral pH. The pure silica was mixed in 2.5 N NaOH by continuous stirring for 10 h with a magnetic stirrer. Concentrated H<sub>2</sub>SO<sub>4</sub> was added to adjust the pH in the range of 7.5 to 8.5. The precipitated silica was washed repeatedly with warm deionized water until the filtrate became completely alkali-free. The precipitated silica was dried at 50 °C for 48 h in the oven.

# Characterization of nano-silica

The morphology of the synthesized silica was examined by scanning electron microscopy (SEM) and energy dispersion X-ray (EDEX) spectrometry by using Advanced

### SEM-EDS (EM-30AX Plus, COXEM).

### Fabrication of the composite

Low-viscosity epoxy resin (Asasin 8205 A) made from bisphenol A and epichlorohydrin and (Asahard 8205 B) hardener made from modified cycloaliphatic polyamide were used from (Asachem (M) Sdn. Bhd, Kajang, Selangor). The epoxy and hardener were used in a 2:1 ratio, as recommended by the manufacturer.

The epoxy resin was poured in a 200 mL beaker. Nano-silica was added to the beaker, drop wise, at concentrations of 5%, 10%, and 15% under mechanical stirring (300 rpm). The mixture was kept under mechanical stirring for 30 min. The hardener was introduced to the mixture, stirred at 300 rpm for another 3 min, and poured into the propylene mold. The mixture in the mold was air dried in an ambient temperature for 7 d and then further cured at 80 °C for 30 min, as recommended by the manufacturer.

#### Determination of water absorption

The specimens were sized to  $25 \text{ mm} \times 25 \text{ mm}$  with a mini band saw. The center line of the square specimens was marked as a cross, using a pencil. Five specimens were obtained for each type of composite and nano-silica. The weight and volume of the specimens were measured, and they were submerged in deionized water for 24 h. The submerged specimens were removed, and any excess water was wiped with a tissue paper. The specimens were remeasured to determine the final weight and volume. The thickness swelling was calculated according to Eq. 1,

Thickness Swelling = 
$$\frac{T_2 - T_1}{T_1} \times 100 \, (\%)$$
 (1)

where  $T_1$  is the thickness of the sample before immersion (mm) and  $T_2$  is the thickness of the sample after immersion (mm). The water absorption was calculated according to Eq. 2,

$$Water Absorption = \frac{W_2 - W_1}{W_1} \times 100$$
<sup>(2)</sup>

where  $W_1$  is the mass of the sample before immersion (g) and  $W_2$  is the mass of the sample after immersion (g).

#### Mechanical tests

The mechanical tests were performed using an Instron universal testing machine (Norwood, MA, USA), equipped with a temperature control chamber. The Young's modulus was obtained from the tangent of the force/deflection curve using 3-point bending tests. Rectangular specimens ( $60 \text{ mm} \times 6 \text{ mm} \times 4 \text{ mm}$ ) with a span-to-width ratio of 8:1 were used. The tests were carried out at a cross-head speed of 1 mm/min.

### **RESULTS AND DISCUSSION**

#### SEM and EDEX analysis

The amorphous and crystalline silica were approximately 200 nm in size and are shown in the SEM and EDEX images. The nano-amorphous silica can be seen in Fig. 1 and the nano-crystalline silica can be seen in Fig. 2. The nano-amorphous silica composed of 54.7% silica (Si) and 44.6% oxide (O), while the nano-crystalline silica composed of 44.9 % Si and 51.7% O.

Elements	Weight (%)
Si	54.7
0	44.6
Na	0.7
Total	100



Fig. 1. The SEM and EDEX of nano-amorphous silica



Fig. 2. The SEM and EDEX of nano-crystalline silica

#### Water Absorption Behaviour

Nano-amorphous silica had little effect on the water absorption and thickness swelling properties of the epoxy composites in the range of 0.08% to 0.09% and 0.19% to 0.22%, respectively. The values were also not significantly different with neat epoxy composite, 0.09% (water absorption) and 0.19% (thickness swelling) as shown in Figs. 3 and 4.

The epoxy composites reinforced with nano-crystalline silica had similar properties to those reinforced with nano-amorphous silica. The silica concentration had no significant effect on the water absorption and the thickness swelling in the range of 0.08% to 0.11% and 0.16% to 0.18%, respectively. The neat epoxy had 0.13% and 0.16% water absorption and thickness swelling, respectively (Fig. 3 and Fig. 4). This indicates that both types of

silica had hydrophobic properties, just as the pure epoxy composite. Overall, the epoxy nano-crystalline silica composite (1.65%) had a significantly higher water absorption compared to the nano-amorphous silica composite (0.20%) (Table 1). The results for thickness swelling showed no significant difference between the nano-amorphous (0.09%) and nano-crystalline (0.10%) composites.



Amorphous silica Crystalline silica

**Fig. 3.** The mean water absorption of epoxy reinforced with nano-amorphous and nanocrystalline silica. The figures in the parentheses are the standard deviations. Means followed by the same letter(s) in the same bar are significantly different at the 0.05 probability level according to the Waller Duncan test.





The water absorption values of the epoxy nano-amorphous composites (0.08% to 0.09%) or the epoxy nano-crystalline composites (0.08% to 0.11%) obtained in this study were very low compared to epoxy reinforced with 15% of lignin (18%), epoxy reinforced with 20% of lignin (16%), epoxy reinforced with 25% of lignin (11%), and epoxy reinforced with 30% of lignin (14%) as reported by Abdul Khalil *et al.* (2011). This is probably due to the hydrophobic nature of nano-silica and the hydrophobic nature of lignin. The hydrophobic character of both nano-silica and epoxy resin would improve the filler

and matrix compatibility during mixing, which it could reduce the amount of voids after curing process. Then *et al.* (2013) found that the incompatibility of polybutylene succinate (PBS) and oil palm mesocarp fibre created the voids and gaps as seen in the scanning electron microscope image.

Droportion	Composite		posite		Cia
Properties	Amorphous	Crystalline	DF	Г	Sig.
Water Absorption (wt%)	0.20	1.65	1	3.82	0.06*
Thickness Swelling (wt%)	0.09	0.10	1	2.32	0.13 <sup>Ns</sup>
Modulus of Rupture (MPa)	62.3	42.6	1	16.01	0.00**
Modulus of Elasticity (MPa)	2587.0	2135.1	1	2.87	0.09*

Table 1. Results of ANOVA on the Ph	ysical and Mechanical Properties
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\* Significant at p < 0.01, \*\* significant at p < 0.001, Ns- not significant at p > 0.1

#### Mechanical properties of epoxy mixed with nano-silica

The static bending of epoxy reinforced with 15% nano-amorphous silica (46.01 MPa) was not significantly different compared to the neat epoxy composite (46.8 MPa), as shown in Figs. 5 and 6.



Fig. 5. Mean MoR of epoxy composites reinforced with nano-amorphous and nano-crystalline silica



**Fig. 6.** The mean MoE of epoxy composites reinforced with nano-amorphous and nanocrystalline silica. The figures in the parentheses are standard deviations. Means followed by the same letter(s) in the same bar are significantly different at the 0.05 probability level according to the Waller Duncan test. The epoxy composite reinforced with 10% nano-amorphous silica (85.03 MPa) had the highest modulus of rupture (MoR), but the value was not significantly different from the composite with a 5% concentration (71.5 MPa). The modulus of elasticity (MoE) increased as the nano-amorphous silica concentration increased. The MoE reached a maximum value for the 10% concentration epoxy composite (3284 MPa), but it was not significantly different for the 15% concentration epoxy composite (3195 MPa).

For the epoxy nano-crystalline silica composites, the MoR increased to 46.4 MPa for the 5% concentration specimen. The highest MoR (47.9 MPa) was seen with the 10% concentration specimen, and then it significantly decreased at the 15% concentration (35.4 MPa). The MoE significantly increased from 2095 MPa at the 5% concentration to 2760 MPa at the 10% concentration. There was no significant difference between the 10% concentration and the 15% concentration (2689 MPa).

In both the amorphous and crystalline composites, the MoR decreased at a 15% silica concentration. This may be due to the intra-molecular adhesion interactions between the filler and the matrix. The composites contain more silica aggregate, which increases the weak zone between the filler particles and the matrix, thereby decreasing the strength of the material. The extra silica content increases the stiffness of composite (Park and Jana 2003). The high number of nanoparticles may have also caused the lower MoR, due to the effect on homogeneity in the crosslinking of the epoxy network. If the interfacial area of the particles is high, then their interaction with the epoxy chain will create less homogeneity in the crosslink density. However, for plant-based fiber composites there is usually limited interaction between the hydrophilic fibers and the hydrophobic matrices. This leads to poor interfacial bonding, limiting mechanical performance and creating low moisture resistance, thereby affecting long term properties. For bonding to occur, the fibers and the matrix must be brought into intimate contact. Wettability is an essential precursor to bonding. Insufficient fiber wetting will result in interfacial defects which can act as stress concentrators (Chen et al. 2006). Fiber wettability has been shown to affect the toughness, tensile strength, and flexural strength of composites (Wu and Dzenis 2006). Physical treatment and chemical treatment can improve the wettability of fibers and improve the interfacial strength (Bénard et al. 2007; Liu et al. 2008; Sinha and Panigrahi 2009; Arun Prakash and Rajadurai 2017; Arun Prakash and Rajadurai, 2016; Arun Prakash and Viswanthan 2019)

Similar to the MoR, the decreased MoE in the epoxy composite reinforced with 15% silica concentration may be due to the agglomeration of nanoparticles in the epoxy matrix (Navaneethakrishna *et al.* 2015). These results agree with those obtained by Satapathy *et al.* (2009) in their study on the influence of silicon carbide (SiC) particles derived from rice husk on the flexural strength of jute/epoxy composites. The flexural strength decreased after dosing SiC particles at 10% and 20% (Satapathy *et al.* 2009). In addition, the decrease maybe due to the poor interfacial bonding between the hydrophobic matrix and the hydrophilic fiber, as explained by Yan *et al.* (2013).

Overall, the epoxy composite reinforced with nano-amorphous silica had a significantly higher MoR (62.3 MPa) and MoE (2587 MPa) than the composite reinforced with nano-crystalline silica, which had a MoR of 42.6 MPa and a MoE of 2135 MPa, as shown in Table 1. This may be attributed to the higher Si content in amorphous silica. The maximum MoR (85 MPa) and MoE (3284 MPa) values of the epoxy composite reinforced with nano-amorphous silica at 15% concentration were higher than the epoxy composite reinforced with 25% lignin, which had a MoR of 74.1 MPa and a MoE of 3020 MPa. The improved MoR and MoE of the epoxy nano-amorphous silica composite compared to the

epoxy lignin composite may be due to the diminished interfacial bonding between the hydrophobic matrix and the hydrophobic lignin (Yan *et al.* 2013). In contrast to the hydrophilic nature of nano-silica, the voids created by poor interfacial bonding between the lignin and the epoxy may have also led to the lower MoR and MoE of the epoxy lignin composite. Akin to wood, this indicates that a higher silica content strengthened the composite.

# CONCLUSIONS

- 1. The physical properties of the amorphous and crystalline nano-silica composites of similar concentrations were not significantly different from one another.
- 2. The thickness swelling was almost identical for the epoxy composite reinforced with nano-crystalline silica and nano-amorphous silica.
- 3. The epoxy nano-amorphous silica composite had significantly higher mechanical properties than the nano-crystalline silica composite.
- 4. Overall, nano-amorphous or nano-crystalline silica dosed at 10% gave the optimum properties for the composite.

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