PHYSICO-MECHANICAL PROPERTIES OF EPOXY RESIN FILLED WITH RICE HUSK PARTICLES

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Abstract

The paper describes an influence of rice husk particles as a filler in a common epoxy resin from the point of view of mechanical properties with the emphasis on the effect of silicon dioxide nanoparticles. The utilised filler has a diameter ranging from 1 to 100 micron. Rice husk particles are a natural composite system composed from organic matrix (cellulose, hemicellulose, lignin) reinforced with amorphous silicon dioxide nanoparticles. This typical composition provides specific parameters of rice husks like strength, hardness and mechanical barrier to the surrounding environment. The optimised composition of the designed composite system was 10 g of rice husk particles added to 1 kg of epoxy resin ChS - Epoxy 520. Rice husk particles in an epoxy resin composite system express an excellent adhesion. The system was evaluated from the point of view parameters like strength, Young modulus, hardness and density. All these parameters show analogous value of the parameters like unfilled epoxy resin. The coefficient of friction shows lower value in case of optimised composition (1 phr) in compare to unfilled epoxy resin. The wear rate (the weight loss of the samples) after adding the filler was increased at 75%. Optimised sample composition was tested for water absorption that was same as in unfilled epoxy resin although the organic particles were present in this epoxy system. The wear rate was positively influenced by the presence of a rice husk filler in a case when other fillers like pigments were added to an epoxy matrix. The described system has a potential to be used in the building industry.

Keywords: composite, rice husk particles, epoxy resin matrix, silicon dioxide nanoparticles

1. INTRODUCTION

Synthetic resins are among materials widely used for impregnation, casting and gluing and in flooring in the construction industry. Some of the most common fillers in this polymer field are inorganic-powder-based particle fillers aimed at decreasing the cost of polymer systems. Unlike thermoplastic polymers, reactoplastics are not generally filled by natural fibrous fillers such as coconut, bamboo and flax fibres. The use of natural particle fillers is usually also intended to save on the polymer matter.

Until recently, rice husks have been overlooked as a material. Their current use is associated with the so-called 3R effect – reducing, reusing, and recycling [1]. Not only are rice husks one of the largest agricultural wastes, globally amounting to approximately 680 million tons per year, but they also posses huge material potential related to the presence of biogenic silicon dioxide nanoparticles in amorphous form. Silicon dioxide nanoparticles, created within the plant as a result of a range of biochemical processes and photosynthesis and owing to the presence of orthosilic acid, are firmly connected to the organic matrix based on cellulose, hemicellulose, lignin, pectin and other substances. Silicon dioxide is excreted by the plant primarily onto the husk surface and provides the plant with many advantages such as reinforcement and barrier functions. The size of nanoparticles detected within the plant is around 20 nanometres [2, 3].

Particles of rice husks, suitably milled and integrated into polymers, may therefore represent an interesting kind of natural filler which is not going deteriorate the original properties of epoxy resin while providing it with new ones to increase its usability in specific fields as well as traditional commercial areas [4, 5, 6].

2. EXPERIMENTAL PART

2.1 Materials

The particle filler used were milled rice husks of Asian rice *(Oryza sativa)* imported from the Khánh Hóa region in Vietnam. The husks have a characteristically high content of silicon dioxide. The size of particles after milling was between 1 and 100 µm, with the particle filler sieved after milling to the required size [4, 5]. A ChS Epoxy 520 epoxy resin (a low-molecular epoxy resin prepared by a reaction between Bisphenol A and Epichlorohydrin without modifying components) hardened by the hardener P11 (Diethylenetriamine) in a mass ratio of 100:11 as specified by the manufacturer DCH - Sincolor, a.s. was used to form a polymer matrix. This resin is used in many branches of industry for impregnation, moulding, casting and gluing and is also suitable for producing cements, adhesives and screed materials [7].

2.2 Methods

The characterization of the input material – rice husks – in terms of describing their surface and chemical composition and localizing silicon dioxide was conducted using a Carl Zeiss Ultra Plus scanning electron microscope with EDX analysis, and the same technique was used to evaluate the size of the particles and assess adhesion between filler particles and the epoxy matrix. Before microscopic analysis, a thin coat of platinum was vapour-deposited onto the specimens. The filler particles were acquired by milling rice husks without prior treatment in a CryoMill Retsch nanomill [4]. The compound (epoxy – rice husk particles) was homogenized after adding the P11 hardener for between 2 and 3 minutes at 40-60 rpm. The compound was subsequently left to harden for 48 hours at regular laboratory temperature and a relative humidity of 50 to 60%. All tests were conducted on hardened specimens after 10 days.
The following tests were selected to determine the value of chosen mechanical parameters: a tensile and compression test (testing instrument P 100 - LabTest II, temperature 23 ± 2 °C, relative humidity approx 60%), a Shore D/15 hardness test, a Charpy impact test – the values determined at standard temperature 23 ± 2 °C, relative humidity approx. 60%, an absorptivity test and the determination of density using the immersion method. The coefficient of friction was evaluated in a CSM ball-on-disc tribometer, with a 100Cr6 steel ball, at a load of 15 N with 20,000 friction cycles at a linear speed of 0.1 m/s under standard laboratory conditions. The wear rate of specimens tested was determined via a tribological measuring method as a specimen’s weight loss after 20,000 cycles. The optimum amount of filler was determined based on the above tribological and wear-rate measurements. Specimens of three selected systems – an unfilled epoxy resin as a standard material, an epoxy resin with 1 phr of filler as a material of optimum composition, and an epoxy resin with 10 phr of filler as an overfilled system for comparison – were used for all subsequent measurements.

2.3 Results and discussion

The microscopic and chemical analysis of rice husks showed that silicon dioxide is present predominantly on the surface of husks. The structure and chemical composition, i.e. the presence of silicon dioxide, of the surface are clearly visible in **Fig. 1** and **2**.

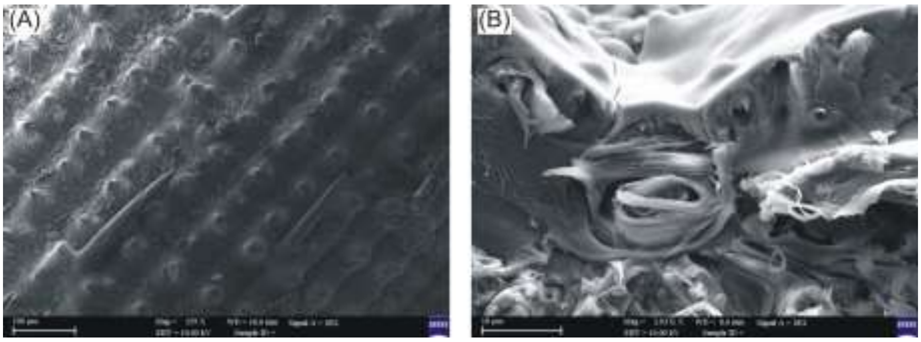

Fig. 1 SEM image of the rice husk surface **(A)**, cross section of rice husk **(B)**.

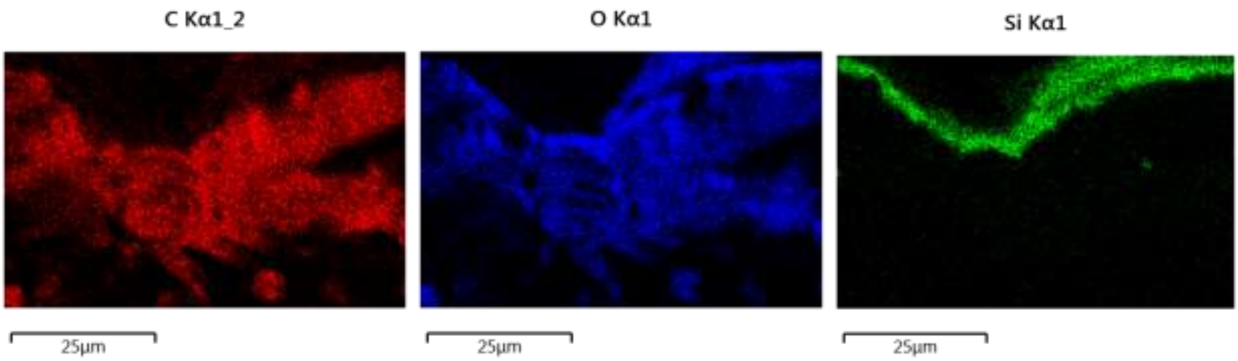

Fig. 2 EDX analysis showing the distribution of elements typical of the composition and structure of rice husks: carbon, oxygen, silicon.

Milled particles used as filler thus contain parts of both organic and inorganic nature. The size of particles obtained through milling in the nanomill is between 1 and 100 micrometres, with the content of silicon being around 12 wt%. The shape of the particles is irregular [4, 5]. Previous experiments showed that the presence of filler particles significantly affects resistance to wear determined by tribological measurements. The explanation for this resistance is that the inorganic phase represented by silicon dioxide acts as a hard particle contributing to a reduced coefficient of friction and increased resistance to wear. In comparison, the organic parts formed by cellulose, hemicellulose and lignin which firmly bind silicon dioxide together are soft and have no effect on mechanical parameters. The explanation for the behaviour of this filler is in keeping with the interpretation of the influence of hard particles in polymers – they transfer part of the load, thus protecting the soft polymer against wear. The adhesion of filler particles to the epoxy resin is good and so is the homogeneity of their distribution within the epoxy resin [6, 8, 9]. For testing mechanical parameters were selected three systems – of a non-filled epoxy resin, an epoxy resin filled optimally for achieving wear resistance and an epoxy resin overfilled with the filler. These three systems selected from a wide range of epoxy filling options are intended to provide evidence of the influence of the amount of filler – none, optimal, excessive – in terms of achieving wear resistance parameters. **Fig. 3** shows the tribological behaviour and wear resistance of tested samples.

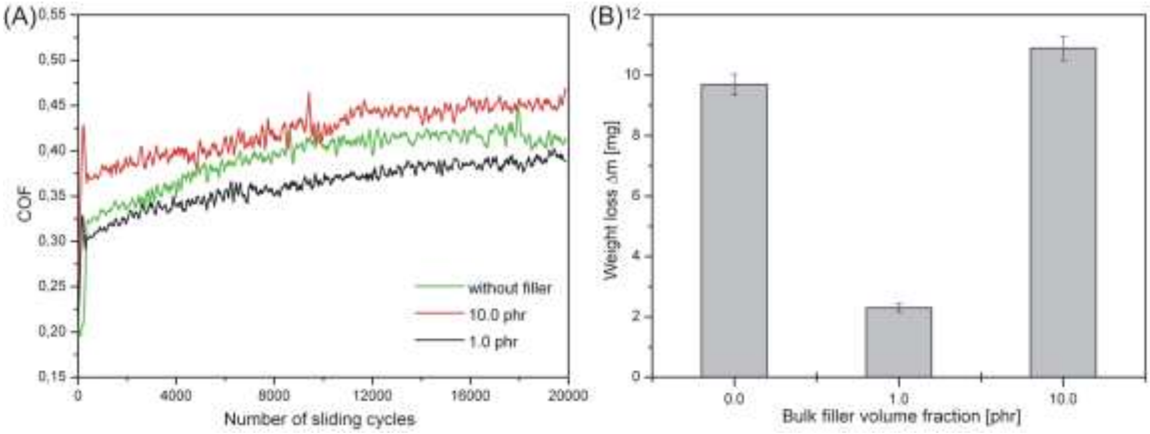

Fig. 3 Graph showing the relationship between: **(A)** the coefficient of friction and the amount of filler under the conditions specified above, **(B)** the amount of filler and its influence on wear rate.

Due to the influence of filler particles on increased wear resistance additional information needs to be provided with regard to their effect on mechanical parameters and also the behaviour of the system in the presence of water. The tensile and compression strength tests show the typical behaviour of the particle filler. When the tensile strength limit was evaluated with respect to the presence of a certain amount of filler, this limit was found to be significantly lower – see **Fig. 4A** – while the limit of compression strength was reduced slightly – see **Fig. 4B**. The tensile strength test results in a slight increase in the modulus of elasticity while the compression strength test slightly reduces it. These differences do not need to be considered as significant in terms of material properties. Although the filler particles used have good adhesion to epoxy resin, mechanical parameters do not increase remarkably because the filler have not the reinforcing character.

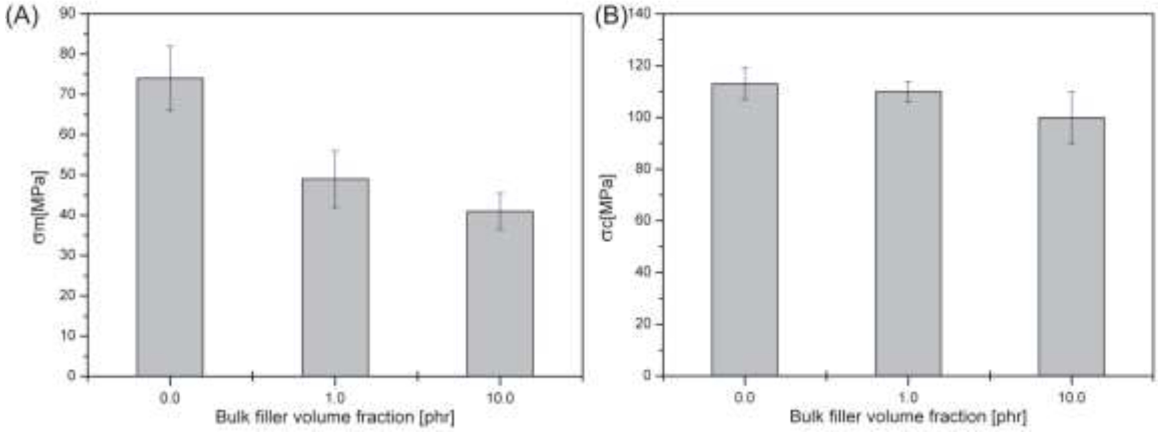

Fig. 4 Graph showing the relationship between: **(A)** the tensile strength limit and the amount of filler, **(B)** the compression strength limit and the amount of filler.

The Shore D/15 hardness values of the systems analysed do not significantly differ – see **Fig. 5A**, not even in the case when dry specimens were compared to ones directly exposed to water – see **Fig. 5B**.

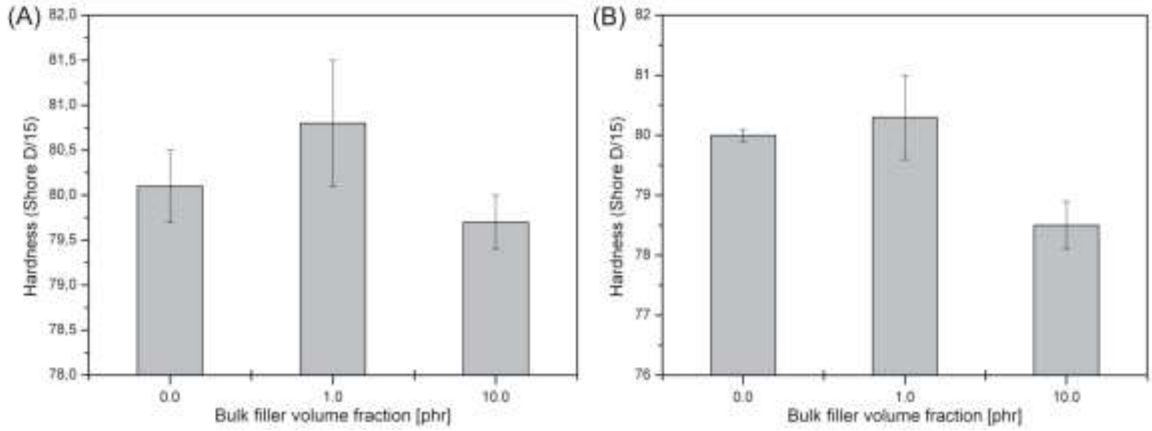

Fig. 5 Relationship between the hardness of: dry specimens **(A),** soaked specimens **(B)**.

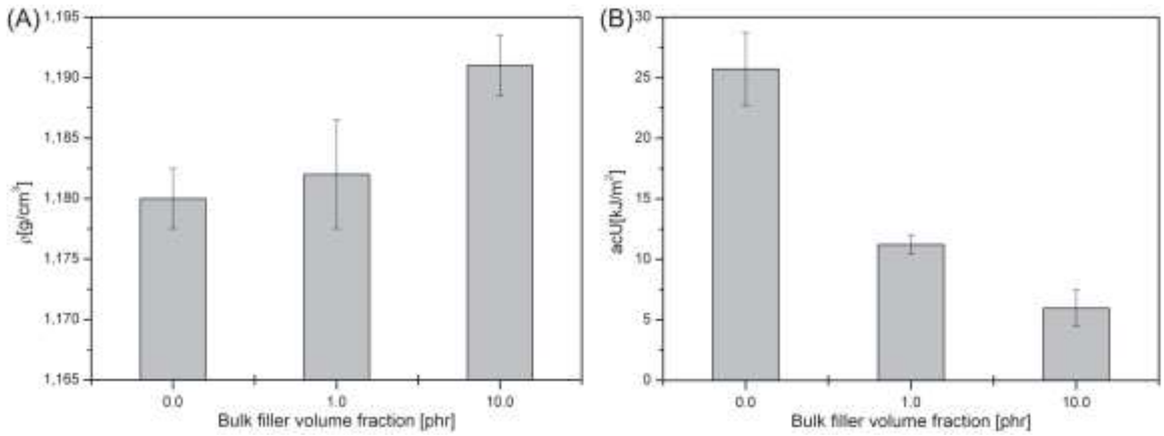

Fig. 6 Graph showing the relationship between: **(A)** the density of each specimen and the amount of filler, **(B)** impact toughness determined using the CHARPY method and the amount of filler.

The density of the composite system with required optimum wear resistance properties increases very slightly – see **Fig. 6A**. However, the impact toughness behaviour changes significantly. The filled systems show remarkably reduced values – see **Fig. 6B**. The influence on this reduction in impact toughness is due to the presence of filler particles which might act as tension concentrators in this case.

With regard to water absorption by the composite system with a particle filler both the optimized composition and unfilled resin have the same properties. The absorption rate is high when filler rates increase to values such as 10 phr – see **Fig. 7A**. The desorption graph clearly shows that water was not completely removed from the system after the specified period of measurement of 6 days (144 hours), neither in the case of the unfilled resin nor in the optimally filled one – see **Fig. 7B**.

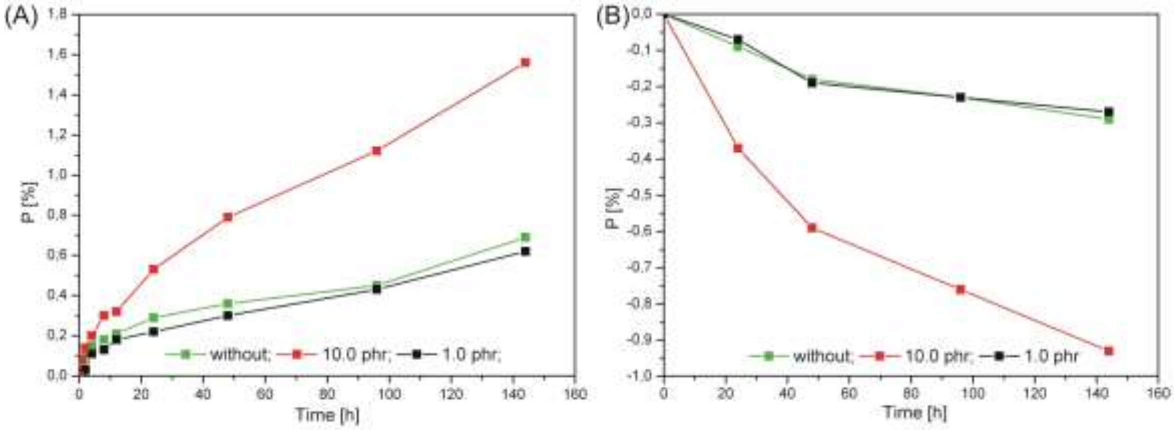

Fig. 7 Relationship between amount of absorbed **(A)**, amount of desorbed **(B)** water in time for each specimen with various amounts of filler.

The use of pigments in epoxy resin leads to a slight increase in the coefficient of friction and reduced wear resistance – see **Fig. 8**. Adding a rice husk filler to the epoxy resin with pigment, on the other hand, increases wear resistance – see **Fig. 8B**. These results were obtained by adding 1 phr of pigment – aluminium bronze and titanium white and a subsequent addition of 1 phr of particles from milled rice husks to the epoxy resin.

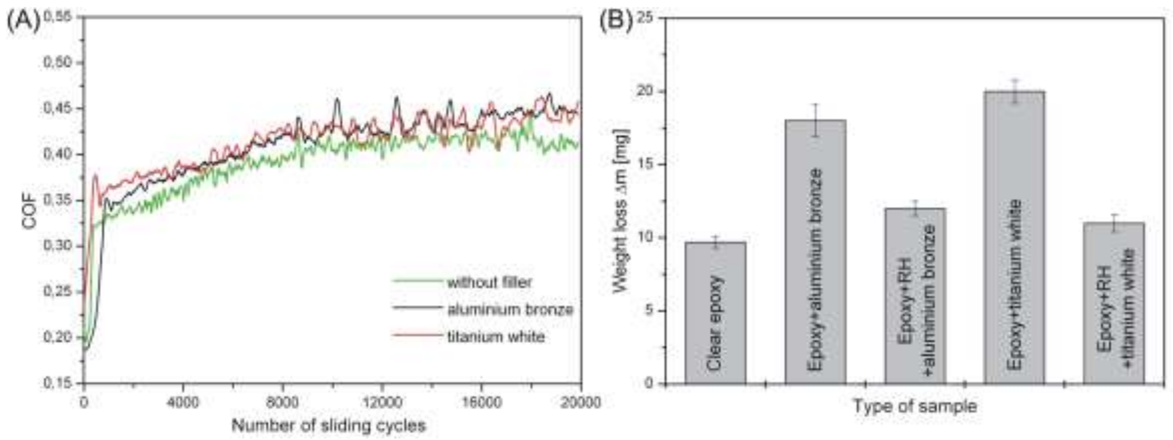

Fig. 8 Influence of pigments on the coefficient of friction in a composite system **(A),** influence of filler and pigments onto wear resistance **(B)**.

3. CONCLUSION

On the basis of the obtained results it was determined that milled rice husk particles between 1 and 100 micrometres in size containing 12 wt% of silicon added to a ChS Epoxy 520 epoxy resin in the amount of 1 phr (1 gram of filler + 100 grams of resin) result in a reduction in the coefficient of friction by approximately 10% and an increase wear resistance by 75%. The overall comparison of the selected mechanical parameters, the coefficient of friction and an increase in wear resistance clearly shows that the content of filler optimized in terms of wear resistance does not lead to significant changes in mechanical parameters with the exception of reduced tensile strength and impact toughness. Particles from milled rice husks behave as a non-reinforcing filler and, in the case of impact toughness, the concentrators of tension. Water absorption in the system with 1 phr of filler was the same as in the unfilled epoxy resin. The filler does not significantly alter the mechanical parameters designed by the producer of the resin while increased wear resistance appears as a new property as a result of the presence of silicon dioxide nanoparticles stored in the organic matrix of rice husks that have good adhesion to the epoxy matrix. The presence of particles of milled rice husks has a positive influence of increased wear resistance even when another phase in the form of selected pigments was present.

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