Experimental description of aging of palm oil kernel shell powder/epoxy composite

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Abstract. Aging of polymeric materials is one of the application limits of these materials. Describing the degradation will help determine the life cycle of the product, describing the degradation process is so important in determining possible application spheres. The paper focuses on the degradation of the matrix composite in the form of epoxy resin and palm oil kernel shells (PKS). Handling bio-waste arising from oil palm processing is a great challenge, material use of these commodities is an interesting alternative to other options. The filler was of irregular particle size up to 100 μm the filler concentration in the matrix was 2.5, 5.0 and 10.0 wt.%. Renewable resources are increasingly promising options for sustainable material engineering. The presence of organic microparticles can optimize some mechanical characteristics and reduce the cost of the resulting material. Microscopy was used to assess interphase interference and microscopic analysis of the surface and particle shape. The contribution showed that the presence of organic particles in the matrix did not significantly affect the decrease of mechanical properties due to degradation in laboratory conditions. Specifically, these were experiments in the climatic chamber where humidity and temperatures varied between +70 °C and -40 °C for 840 hours. The decrease in shear strength on steel adherence reached 3.99 MPa in the unfilled resin and 5.77 MPa (10% PKS) in the palm kernel shell resin.

1. Introduction
Biocomposite materials are an interesting alternative to conventional material engineering [1]. A composite of natural fibers can be considered as a very perspective group [2]. According to Ku et al. [3], natural fibers in composite systems make materials attractive to researchers, engineers and scientists, especially due to their low cost, relatively good mechanical properties. A similar economic benefit can be expected for composites with natural particulate fillers, but the optimization of mechanical properties will be different and will interfere with other characteristics. An example may be the work of authors’ colleagues [4] describing optimization especially in the field of shear strength. In composite systems, secondary raw materials arising from the processing of natural resources can also be used. An example may be palm oil, which holds the dominant global position in the production of vegetable oil.
The Asian region is one of the largest palm oil producers in the world. Both Indonesia and Malaysia are the first in the volume of production, and these countries are also responsible for handling agricultural waste generated by palm oil production. Ramli [5], in his analysis, states that production growth in Malaysia is steadily rising (by 2020 expects 5 million hectares of palm oil trees). From the point of view of the recovery of waste generated during processing, this is still a current problem. On the technological lines processing the palm oil fruits, there is an accumulation of further usable
products. The first product is cellulose fibers, which can also be used in the field of material engineering. The second product is the fruit shell. They either use energy applications in different forms or can be integrated into the field of material engineering. Materials utilization in composite systems in interaction with polyester resin is described by Nabinejad et al. [6], another way to use PKS is, for example, application to rubber [7]. The experiment compares the shear strength of the overlapped bodies using PKS and the resin itself while describing the aging of the test bodies in the climatic chamber.

2. Materials and methods
In the experiment, fruits from palm oil from processing lines in Sarawak, Borneo were used (see Figure 1).

Figure 1. An illustrative example of oil palm fruit handling: A-fruit after harvest, B-fruit processed on a technological line (shell and core), C-shells after core removal, D-prepared particles, E-core, that is pressed on technological line.

Shells were processed with a knife mill at a speed of 20,000 rpm·min⁻¹, after which the particles were dried in an oven at 105 °C. Subsequently, individual fractions of PKS were prepared through analytical sieves. After the preparation of the individual fractions, composite systems were made such that the epoxy resin was mechanically mixed with PKS. The resulting concentration of PKS in the matrix corresponded to 2.5, 5.0 and 10.0 wt.%. For the description of shear strength on steel sheet S235J0, the standard CSN EN 1465 was used. The roughness parameters of the steel sheets prior to application of the composite mixture were measured by the touch grinder. The conditions defined in CSN EN ISO 914 were used to simulate the aging of the material in the climatic chamber. The total degradation time was 840 hours, which is 35 cycles. One cycle of degradation corresponded to 24 h. In one cycle, the bodies were tempered to 70 °C at 90% humidity for 16 hours, then subcooled to -40° C for 3 hours and then returned to + 70 °C at 50% humidity. For the evaluation was used ANOVA.

3. Results and discussion
Optical analysis of particle size and particle shape was performed using microscopy, and the surface of the particles was then analyzed on an electron microscope (Figure 1-a). The particle surface (Figure 2-b, c) is covered by pores that are responsible for increased water absorption [8]. It should also be noted that the surface of the shell is covered with fibers, according to [9] the fibers are up to 30% of the surface, as can be seen from Figure 1 c. After the optical analysis, the particle size was determined as follows: 58 ± 29 μm. Optical surface analysis of PKS confirmed that microscopic structure even with the presence of pores is very similar to that of other types of fruit shells, e.g. coconuts.
The shear strength is influenced by the roughness of the surface of the material (Ra = 2.7 ± 0.6 μm and Rz = 11.9 ± 3.2 μm). The shear strength of the resin without filler was 11.75 ± 0.85 MPa. With the addition of filler, shear strength was increased by 1.18 MPa (2.5% PKS), 0.70 MPa (5.0% PKS) and 1.13 MPa (10.0% PKS) - adhesive type of failure. An important aspect affecting the resulting mechanical properties of composite systems is the interfacial interaction. In the case of oilseeds, it is necessary to point to a higher proportion of oils in plant residues, which can adversely affect the formation of interfacial phases. For this reason, the mutual phase interaction was carried out by optical analysis (see Figure 3), which confirmed the corresponding interfacial interaction.

The course of degradation compared to the filled and non-filled resin is shown in Figure 4. The presence of PKS fillers did not significantly increase the rate of degradation. After the degradation cycle (840 h), the non-filler resin strength dropped to 7.76 ± 0.83 MPa, PBS composite with 2.5% filler to 7.93 ± 1.21 MPa, 5.0% to 7.98 ± 1.15 MPa and 10.0% to 7.52 ± 1.46 MPa - adhesive type of failure. Conclusions of the experiment confirm the possible use of agricultural waste in the form of material engineering, as described by a number of authors [2, 8, 10, 11].
Figure 4. Course of degradation of the non-filled and filled resin (interval 0-840 hours - left), the test body before degradation and corrosion of the steel body after the degradation - after 840 hours (right).

4. Summary
The article describes a possible alternative of the use of PKS, which could contribute to measures leading to the exploitation of by-products of agriculture - sustainable development. It is an environmentally friendly way of using materials to prevent environmental pollution and minimize the consumption of primary raw materials. The disadvantage of the biological microparticles used may be an increased proportion of vegetable oil in these agricultural residues and a tendency to absorb water. The performed experiment did not show an increase in the rate of degradation of the test bodies with PKS comparing to the unfilled resin. At the same time, the experiment has shown that the presence of PKS slightly increases the shear strength of the assemblies. Optical electron microscopy analysis confirmed the good wetting of PKS with epoxy resin.

5. References
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Acknowledgments
This paper has been made with the assistance of the grant IGA TF CZU.