Investigation on Polymer Composite Materials Wear Reinforced by Microparticles of Jatropha Curcas L. Waste

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Abstract. The subject of the paper is a research on the wear resistance by friction against loosely fixed abrasive particles on a polymer composite material reinforced by waste microparticles from a pressing process of Jatropha curcas L. seeds with concentration from 5 to 20 wt.%. The seed pressing waste was used in form of Whole seeds cake (WSC), Seed shells (SS) and Seed kernels cake (SKC). The waste, which is difficult to further utilize for its inability to use as feed, arises at the pressing process of Jatropha Curcas L. seeds. A matrix of composite materials was epoxy-based. The wear tests were performed by the device Tester T-07 according to GOST 23.208-79. An addition of the filler significantly increased the wear resistance by friction against a rubber wheel with loosely fixed abrasive particles of sand. The wear resistance was increased up of 69% to 82% at tested polymer composite systems against matrix (resin). Composites materials with the filler Whole seeds cake (WSC) achieved the best results.

1. Introduction

The composite materials find applications in various field of industry production, where is necessary to consider not only a strength but also the wear resistance [1, 2, 3, 4, 5, 6, 7]. The wear resistance has own specification and understanding tribological behaviour of composite materials is important [8, 3]. For improvement the wear resistance various fillers based on micro and nanoparticles have been historically used [9].

A particle of SiC, Al₂O₃, ballotine, copper, aluminium and short carbon fibre, glass fibre etc. could be an example [4, 10, 11, 12, 13, 14]. An alternative filler based on natural materials, e.g. wood flour, microparticles from egg shell, nut shells, fruit stones etc. are used currently [15, 16, 17].

Use of the filler in a composite material field has three main reasons. The first one is a cost reduction, the second one is an increase of mechanical properties and use of the filler for improvement of the wear resistance. At the tribological behaviour of the composite material it is necessary to
consider the wear type on a product surface and interactions between a matrix and reinforced phase inside of the composite structure [18, 5]. An inappropriate interaction between the matrix and the filler leads to increased wear of the material which is caused by a loose of the material, i.e. the mass loss [19].

The third one is the use of waste from agricultural commodities production, which is renewable source. One of a possibility is the material use of waste from Jatropha Curcas L. seed pressing, i.e. Jatropha Oil Cake. This waste proved its positives at tribological tests ball-on-flat with reciprocating motion with frequencies of 5 Hz and 10 Hz at polymer composite materials [20]. Main use of seeds from the plant Jatropha Curcas L. is oil production by pressing [21]. Oil cakes are secondary products after mechanical oil pressing which proved their strengths in practice [6]. The pressed oil and oil cakes are not eatable. For this reason, the secondary product is not suitable for farm animals feeding. At next treatment of Jatropha oil cakes, the filler arises which can be used in polymer composites. The example of the effective use can be seen e.g. in research by Shivamurthy [22] and Mohan [23], who proved the effectivity of the filler from oil cake in powder form e.g. improvement of tribological properties.

The aim of the research is an evaluation of the polymer composite wear resistance with the matrix based on the epoxide and the reinforced phase based on waste from Jatropha Curcas L. seed treatment, i.e. from oil pressing in form of Whole seeds cake (WSC), Seed shells (SS) and Seed kernels cake (SKC).

2. Materials and methods

The particle polymer composite with a continuous phase in a form of a two-component epoxy adhesive Lepox 1200 and a discontinuous phase (reinforcing particles) in a form of waste from pressing process of Jatropha Curcas L. seeds in a form of Whole seed cakes (WSC), Seed shells (SS) and Seed kernel cakes (SKC) was the subject of the experiment. A distribution of the input raw material for a preparation of the filler came out from the secondary product at oil production.

WSC, SS and SKC were desiccated at the temperature 105 ± 5°C for 24 hours and then powdered by multi-stage crushing machine. This input material was sorted by the sieve analysis. For research a smallest fraction was used which fell into sieve with size 100 µm of mesh. The size of particle fillers WSC, SS and SKC were measured by means of an image analysis with programme Gwyddion and images from electron microscope (SEM). The size of WSC was 72.28 ± 38.5 µm, size of SS was 62.42 ± 28.6 µm and size of SKC was 68.56 ± 42.8 µm.

Test samples for the wear resistance evaluation according to standard GOST 23.208-79 (Ensuring of wear resistance of products. Wear resistance. Testing of materials by friction against loosely fixed abrasive particles) were made in the form from silicone rubber Lukopren N 1522. The test samples had dimension 25 x 25 x 5 mm. The composite was made from the epoxide and the reinforcing phase with concentration 5, 10, 15 a 20 wt%. The silicone form was filled by composite mixture and cured for 24 hours at laboratory temperature.

The evaluation methods of composite materials with the filler WSC, SS and SKC were based on self-comparison of the wear resistance according to standard GOST 23.208-79. The test of the wear resistance was measured according to standard GOST 23.208-79 i.e. on device with rubber wheel (fig. 2 A). The worn test samples with the characteristic wear track are shown in fig. 2 B, C, D. The float sand Otava with particles of the size 0.2 to 0.315 mm fell between the test sample and rubber wheel. The test samples were load by 680 g. The test samples were cleaned with compressed air before and after the test. The mass loss was measured by weighing-machine KERN ABS with tolerance 0.1 mg. The test samples were measured at 100 turns of the rubber wheel. For each measuring three samples were used. The test samples after wear test are shown in fig. 2 B, C and D, where a typical relief of the worn surface is noticeable.
3. Results and discussion

By the electron microscopy the shape, the size and the texture surface of the fillers Whole seeds cake (WSC), Seed shells (SS) and Seed kernels cake (SKC) were observed, see fig. 3. The fillers WSC, SS and SKC are different among themselves in the shape characteristic, especially Seed kernel cakes have very rugged surface composed of many segments.

The process of the matrix wear (resin, without the filler) depending on the wear track is shown in fig. 4. The comparison of composite material results in fig. 5, 6 and 7 points on the significant influence for the wear resistance improvement of polymer composites. The average value of the worn matrix (resin) is $51.99 \pm 6.95$ mg. The composite materials reported much smaller values of the wear, i.e. the mass loss was in interval from 9.49 to 15.81 mg. The results comparison is evident from fig. 8.

The process of the wear depending on the track is shown in fig. 5, 6 and 7 which present a cumulative mass loss depending on a trajectory. The results point on a linear increase of the mass loss.
of tested samples. For the relevant evaluation it is necessary to determine the strength (intensity) of the linear dependence which is the aim of correlation analyses.

![Figure 4. Matrix wear process (resin)](image1)
![Figure 5. Wear process of composite material with filler Whole seed cakes (WSC)](image2)
![Figure 6. Wear process of composite material with filler Seed kernel cakes (SKC)](image3)
![Figure 7. Wear process of composite material with filler Seed shells (SS)](image4)

Each filler concentrations of composite materials were tested among themselves according to ANOVA F-test in significance level 0.05. The results of the statistical comparison are shown in fig. 8. Composite materials showed less dispersion than the matrix. For null hypothesis $H_0$ the status was marked when there is no statistically significant difference among the data sets being compared in terms of their mean values: $p > 0.05$. The hypothesis $H_0$ was not confirmed at composite materials with filler Seed shells (SS) and Seed kernel cakes (SKC), i.e. there is the significant difference ($p = 0.000$) among the tested materials depending on the different filler concentrations in the significance level 0.05. The hypothesis was confirmed on composite with filler Whole seed cakes (WSC), i.e. there is no difference on the wear resistance among tested concentrations ($p = 0.098$).
The best and at the same time balanced results were achieved on the composite material with the filler Whole seed cakes (WSC) which contained hard particles of Seed shells (SS) and soft particles of Seed kernel cakes (SKC) which contained increased percentage of oil.

![Image](image.png)

**Figure 8.** Statistical comparison of measured results of composite materials

The surface of the worn composite material with the filler Whole seed cakes (WSC) is shown in fig. 9. In fig. 9 A the texture of the worn surface at MAG 5.00 kx is noticeable. In fig. 9 B (MAG 1.60 kx) the detail on a delamination of larger particle from the matrix is noticeable. The delamination of larger particles decreases the wear resistance. In fig. 9 C larger particle with good adhesion force is visible which did not allow it to release from the matrix. Whole seed cakes (WSC) contain soft particles of a core and hard particles of a peel.

![Image](image.png)

**Figure 9.** SEM images of worn surface according to GOST 23.208-79 of composite material with Whole seed cakes (WSC)

The worn surface of the composite material with the filler Seed shells (SS) is shown in fig. 10. Seed shells (SS) are hardener than Seed kernel cakes (SKC). On the worn surface a significant groove was examined - see fig. 10 A (MAG 500 x) with detail in fig. 10 B (MAG 500 x) and C (MAG 3.26 kx).
Figure 10. SEM images of worn surface according to GOST 23.208-79 on composite material with filler Seed shells (SS)

The worn surface of the composite material with the filler Seed kernel cakes (SKC) is noticeable on fig. 11. This filler contains the largest percentage of residual oil from pressing process and contains smaller particles, see fig. 3 C. In fig. 11 A (MAG 1.50 kx) a typical relief of the worn surface is obvious. At the filler Seed kernel cakes (SKC) larger filler cluster was pulling out, see fig. 11 B (MAG 5.00 kx), which is subsequently applied to the surface in the direction of a disc rotation due to the pressure force of the rubber disc during the wear process, see fig. 11 C (MAG 100 x).

Figure 11. SEM images of worn surface according to GOST 23.208-79 on composite material with filler Seed kernel cakes (SKC)

The research results confirmed the fact that the filler addition into the thermosetting matrix based on epoxide influenced the mechanical properties of the composite [13, 14, 24, 25]. The significant factor is the type and the dimension of the filler [10]. The conclusions of many authors regarding the possibility of effective use of the filler based on waste from agricultural products processing in the area of polymer composite materials were certified [6, 15, 16, 17, 19].

4. Conclusions

The polymer composite materials based on waste from pressing process of Jatropha Curcas L. seeds in the form of Whole seed cakes (WSC), Seed shells (SS) and Seed kernel cakes (SKC) with concentration from 5 to 20 wt.% proved the significant wear resistance by friction against the rubber wheel with loosely fixed abrasive particles of sand according to GOST 23.208-79.

The wear resistance of polymer composite test samples was increased to the matrix (resin) up of 69% to 82%. The best results reported the composite material with the filler Whole seed cakes (WSC). The statistical analysis of this filler proved that the wear resistance was not directly dependent on the filler concentration in interval 5 to 20 wt.%. This filler reported also the least dispersion of results. These results are probably caused by microparticles combination of soft filler Seed kernel cakes (SKC) and hard filler Seed shells (SS).

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