Tribological Analysis of Jute/Coir Polyester Composites Filled with Eggshell Powder (ESP) or Nanoclay (NC) Using Grey Rational Method

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Abstract: The wear performance of jute/coir unsaturated polyester composites, filled with eggshell powder (ESP) and nanoclay (NC), were examined, concentrating on two measured parameters, coefficient of friction (COF) and wear rate (WR). To assess the possibilities of this material, a Taguchi study, based on grey relational analysis (GRA), was carried out, based on three testing parameters of the wear performance, load (10, 20, and 30 N), speed (100, 150, and 200 rpm), and sliding distance (30, 40, and 50 m). The material showed promising characteristics especially at high load, low speed, and high sliding distance. When comparing the respective influence of the three different parameters, the speed proved to be the most critical, this suggested the possible application of the biocomposite only for very low values of it. On the other hand, it was also elucidated that the presence and interfacial adhesion of the two fillers considerably hindered the formation of ploughing during wear test, despite the fact that degradation might be continuous and critical as far as loading progresses.

Keywords: coir; jute; eggshell powder; nanoclay; wear rate; coefficient of friction; Taguchi; grey relational analysis

1. Introduction

Natural fiber reinforced polymer composites have received considerable attention in recent years in a variety of applications, including numerous industrial applications, for example, in the automotive, furniture, and construction fields [1,2].

An issue that is essential for the aforementioned sectors is the resistance of natural fiber composites to wear and friction with hard mechanical bodies they might come into contact with. The abrasive contact of the friction pair elements causes wear and tear, which in turn reduces the operational efficiency of components. The effects of friction on material wear caused by tribological processes, such as abrasion, cracking, and crushing of particle materials, adhesion of the mating element surface, and tribo-chemical processes, have been widely investigated [3–5]. One of the most followed procedures to ensure an improvement of tribological performance of metals, such as aluminum alloys, is the introduction of harder particles into them, for example, typically graphite [6], or ceramic particles, such as alumina, zirconium oxide and nitride [7], boron carbide [8], etc.
Abrasion resistance is a research theme that is central to the development of polymer composites, such as fiberglass or carbon fiber composites, as a possible replacement in terms of lower weight and comparable specific performance for metals [9]. When passing to natural fiber composites (NFCs), some lignocellulosic fibers are able to provide a sufficient hardness and resistance to abrasion to represent a potential reinforcement for NFCs intended for tribological use. This is, for example, the case for jute and coir. In particular, the use of jute has been involved in hybrid polymer composites with glass and bamboo fiber [10] or with hemp and flax fibers [11], for abrasion resistant purposes. In the case of coir, its durability allowed even application in concrete as the replacement for coarse aggregates [12]. More recently, the use in phenol–formaldehyde resins as automotive brake friction materials investigated the respective influence of slag and coir fibers [13].

The introduction of ceramic materials, such as silicoaluminates (e.g., nanoclay) or calcium carbonates (also in the form of eggshell powder, a waste product from the food industry) in natural fiber composites has become a quite common practice to improve shear resistance and prospectively provide durability under abrasion phenomena. As an example, nanoclay has been demonstrated to improve the waterjet cutting precision into jute/polyester composites [14] and a mixture of jute and eggshell powder has been proposed as a filler for cement–paper composites for automotive dashboards [15]. As regards coir, its use in an epoxy matrix together with nanoclay allowed obtaining a better stiffness with no detriment to its ductility [16]. In particular, eggshell powder (ESP) was used with coir to provide an improved compressive strength with immediate reduction of the tensile one [17]. Furthermore, ESP represents an example of post-consumer waste, which is widely available and was proven to be effective, even when introduced in small amounts, to increase the wear resistance of matrices, including metal ones (aluminum), and polymer ones (polyethylene) [18].

The objective of this work is to broaden the field of application of jute/coir hybrid composites, whose interest lies particularly in being produced using two diffuse, complementary, and lignin-rich fibers, well rooted into applications in different sectors, which suggested extensive studies on their properties [19]. More specifically, to try to optimize the presence and find the optimum combination of these four components, namely coir and jute fibers, nanoclay, and eggshell powder, with markedly different yet possibly synergic characteristics, a Taguchi approach is proposed. The idea is more specifically to determine the significance of process parameter interaction effects [20,21]. As regards tribology, the Taguchi method is useful to obtain indications on the respective influence of different control parameters on wear resistance and has been used already on a number of more traditional composites, such as aluminum ones, filled with titanium carbide particles [22].

The Taguchi approach consists of three consecutive parts: design, analysis, and optimization. The optimum process designs are determined by analyzing the setup for the study outcomes using a signal to noise (S/N) ratio; this is obtained by dividing the mean (signal) by the standard deviation (SD) (noise), with the idea that reduced S/N ratios (“the lower the better”, or LB) are preferred over higher S/N ratios (HB), and nominal S/N ratios are preferred over lower S/N ratios (NB). When it comes to reducing wear, the LB feature is a necessity. Dry sliding wear behavior of composites has been successfully studied using this method. These techniques are aimed at bettering the design of industrial processes [23,24].

As initial sets of data are normally insufficient, unfinished, and provide ambiguous information, Deng [25] proposed the grey system hypothesis to elucidate the inadequate information link between these data. The first step is S/N ratio normalization, followed by grey normalization. The final step is to calculate the grey rational coefficients to illustrate the correlation between the desired and actual experimental results using normalized data. Based on the determined grey relational grade, the overall grey relational grade is generated by averaging the grey relational coefficient corresponding to each performance feature. As a result, optimization of a single grey relational grade replaces optimization
of a complex multiple performance characteristic. The process parameter level with the highest grey relational grade is the optimum level.

The specific purpose of this study was to optimize tribological test settings to reduce jute coir/ESP1.5%/NC1.5% friction and wear. In addition, ANOVA (analysis of variance) was used to forecast the impact and each process parameter’s significance, as well as their interface, on the tribological characteristics of jute coir/ESP1.5%/NC1.5%. Finally, a confirmation test was run to ensure that the optimal combination of process parameters determined by the analysis was correct.

2. Materials and Methods

2.1. Materials

The two fibers that have been selected on the local market for their plentiful availability are jute and coir, which have both been obtained in the form of mats with an areal weight of 220 g/m². The matrix was fabricated using unsaturated polyester resin, methyl ethyl ketone peroxide (MEKP) catalyst and cobalt naphthenate (accelerator), which were provided by Aiswarya Polymers in Coimbatore, Tamil Nadu, India. In total, 100 g of resin were cured in the presence of 1.5 milliliters of MEKP and 1 milliliter of cobalt naphthenate. For chemical treatment, a 5% solution of sodium hydroxide (NaOH) was applied to the fibers for 12 h.

Two different fillers were also used to reinforce the matrix and compare their respective effectiveness to the purpose. In particular, the two fillers were:

- Eggshell powder (ESP), from eggs obtained at local pastry shops, heat treated at 450 °C, as indicated in [26], as a suitable method for the removal of protein membrane. The powder was then reduced in size by ball milling, using a PM100 (Retsch, Haan, Germany) planetary ball miller, at a rotating speed of 200 rpm, to an average granulometry down to a fineness between 20 and 100 microns;
- MMT K10 (Montmorillonite) nanoclay (NC) with a surface area of 220–270 m²/g supplied by Sigma-Aldrich, Bangalore, Karnataka, India.

2.2. Chemical Treatment of the Fibers

Detergent washing, alkali treatment, and dewaxing enhanced the interfacial adhesion between hydrophilic coir jute fabric mats as well as a hydrophobic polyester matrix.

- Dirt was eliminated from the mats by soaking them in a 5% detergent solution for 10 h at room temperature (liquid hand washing detergent with 5–15% non-ionic tensioactive chemicals);
- Dewaxing was achieved by immersing the cloth in a 5% ethanol (C₂H₅OH) solution for one hour at room temperature, eliminating all traces of pectin and waxes;
- To remove loose fibers and harden the fiber mats’ surface, they were immersed in a 5% sodium hydroxide (NaOH) solution and dusted using a 6% acetic acid (CH₃COOH) solution.

Each step was followed by a rinse phase in clean water, taking several minutes, and a natural drying process for 2 h.

2.3. Production of the Laminates

The compression molding method was used to manufacture polyester composites. The composites containing a total combination of 3 wt.% of NC/ESP filler (1.5 wt.% of each one) were produced using a mold with dimensions of 300 × 300 × 3 mm³; the above combination offered better mechanical properties in a previous work [27]. Jute and coir fiber mats were also cut to a size of 300 × 300 mm.

To ensure the mixing of the resin with NC and ESP, stirring at a velocity of 500 rpm for one hour at room temperature was performed. A vacuum oven was then used for degassing the mixture before combining it with the catalyst and accelerator. When the molding process was completed, a wax coating was applied on the mold box to ensure easy and complete demolding. After degassing, the wax-coated mold box was brushed with
a small amount of mixed matrix to avoid composite adhesion and poor impregnation. A matrix mixer was also poured between each mat, as these were carefully placed in the mold one by one, until a total of three layers of jute and three layers of coir was reached. The structure is reported in Figure 1. The remainder of the mixture was poured over the fiber mats, and the mold box was sealed. In practice, the total amount of reinforcement in the composite was equal to 40% fibers, of which 20% was coir and 20% jute. For consolidation, the pressure was applied to the mold box and maintained at room temperature for up to 6 h. After compression molding, the composite plates were removed, and the solid composite plates were exposed to post curing at 80 °C for up to 4 h.

Figure 1. Scheme of the laminates lay-up before pressure consolidation.

The fabricated composites were taken out from the mold and cut into the recommended dimension required in ASTM G99-17 standard for tribological testing. The test was carried out by a pin-on-disc wear tester. Figure 2 shows the experimental setup for a pin-on-disc wear tester (Make: Magnum Engineers, Bangalore), using a 5 mm pin on a 55 × 10 mm disc.

Figure 2. Experimental setup for pin-on-disc wear tester.
The two wear parameters measured were, in particular, wear rate (WR), obtained by the ratio of volume loss (VL) in m$^3$ divided by sliding distance (SD) in m, and coefficient of friction (COF), obtained by the resistive force of friction (Fr) divided by the normal force (F) that pushes the two forces together.

2.4. Control Factors and Matrix of Experiments

Multiple attribute decision making (MCDM) aims to evaluate the combination of different objectives in order to optimally resolve the issue; in this case, the specific goal was to clarify which would be the most appropriate mix of values for the three factors, load (A), speed (B), and sliding distance (C), resulting in the most desired traits for the application envisaged for the laminate produced, namely for automotive washers and spacers. Here, the grey relational analysis (GRA) approach was utilized to predict the influence of restricting criterion values on the final conclusion, to check the calculations, and to contemplate the possibility of the method’s evaluations being flimsy due to the unique nature of the real data.

As a result, a three-level L9 orthogonal array with three parameters (load in N, speed in rpm, and sliding distance in m) was utilized to optimize the variation and achieve a lower wear rate and coefficient of friction (COF), allowing the automobile component to be accommodated more easily. The control parameters for three levels of GRA are presented in Table 1, while the L9 matrix was exposed in Table 2.

Table 1. Factors and levels for experimental work.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Level 1</th>
<th>Level 2</th>
<th>Level 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load (N)</td>
<td>10</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>Speed (rpm)</td>
<td>100</td>
<td>150</td>
<td>200</td>
</tr>
<tr>
<td>Sliding distance (SD) (m)</td>
<td>30</td>
<td>40</td>
<td>50</td>
</tr>
</tbody>
</table>

Table 2. Sample codes, variations (Var.) and parameter values for experimental work.

<table>
<thead>
<tr>
<th>Code</th>
<th>Var. 1</th>
<th>Var. 2</th>
<th>Var. 3</th>
<th>Load (N)</th>
<th>Speed (rpm)</th>
<th>Sliding Distance (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>10</td>
<td>100</td>
<td>30</td>
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<td>1</td>
<td>2</td>
<td>2</td>
<td>10</td>
<td>150</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>3</td>
<td>3</td>
<td>10</td>
<td>200</td>
<td>50</td>
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<td>2</td>
<td>20</td>
<td>100</td>
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<td>2</td>
<td>2</td>
<td>3</td>
<td>20</td>
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<td>50</td>
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<td>2</td>
<td>3</td>
<td>1</td>
<td>20</td>
<td>200</td>
<td>30</td>
</tr>
<tr>
<td>7</td>
<td>3</td>
<td>1</td>
<td>3</td>
<td>30</td>
<td>100</td>
<td>50</td>
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<td>3</td>
<td>2</td>
<td>1</td>
<td>30</td>
<td>150</td>
<td>30</td>
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<td>3</td>
<td>2</td>
<td>30</td>
<td>200</td>
<td>40</td>
</tr>
</tbody>
</table>

3. Results

3.1. Optimization of the Composite Performance Using GRA

To improve the mechanical properties, the grey inspection used involved the following steps:

Step 1: control factors are identified (load: A, speed: B, sliding distance: C), as well as their levels of control.
Step 2: the appropriate orthogonal array (OA) for experimental analysis is defined.
Step 3: the experiments to determine the responses are carried out using pin-on-disc setup.
Step 4: the S/N ratio values for the experimental values of wear rate (WR) and coefficient of friction (COF) are calculated.
Step 5: the aforementioned S/N ratio values are normalized.
Step 6: the grey normalization and grey coefficients are calculated.
Step 7: the grey grade is measured, and the alternatives are ranked.
Step 8: the analysis of variance (ANOVA) is performed.
In this study, the Taguchi technique, a specific tool for designing process parameters, was utilized. The Taguchi technique is a method for determining the best set of design parameters in a systematic and efficient manner. This is a quality assessment of the obtained experimental results. The signal-to-noise (S/N) ratio was used in this study. Taguchi uses the signal-to-noise ratio notion from noise to multi-factor studies. The S/N ratio is a statistic feature that compares the mean and variance of two data sets. S/N ratios can be classified into three categories: the lower the better (LB), the higher the better (HB), and nominal best (NB). In this case, the principal goal is to reduce pin-of-disc wear of rate and the coefficient of friction. As a result, the lower the better type (LB) S/N ratio was used, which was obtained by Equation (1):

\[
S/N \text{ ratio} = -10 \log_{10} (y^2)
\]

where \(y\) are the values of the single experiments. For every set of conditions, three samples have been tested, on which WR and COF have been measured, then the average values, on which Taguchi experiments have been carried out, and their respective standard deviation have been measured.

Following this, the S/N ratio is normalized with the criterion of “the lower, the better”, therefore assuming that 1 corresponds to the lowest, then the best value, and 0 corresponds to the highest, hence the worst value.

The normalization can be obtained by Equation (2):

\[
x_i(k) = \frac{\max y_i(k) - y_i(k)}{\max y_i(k) - \min y_i(k)}
\]

where \(k\) is the measured parameter, and \(y_i\) is the signal-to-noise ratio of every single experiment. The results are exposed in Table 3.

**Table 3.** Data from wear tests and signal-to-noise ratio (absolute and normalized).

<table>
<thead>
<tr>
<th>Load (N)</th>
<th>Speed (rpm)</th>
<th>SD (m)</th>
<th>WR</th>
<th>COF</th>
<th>S/N</th>
<th>WR</th>
<th>COF</th>
<th>Normalized S/N</th>
<th>WR</th>
<th>COF</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>100</td>
<td>30</td>
<td>101 ± 35</td>
<td>0.35 ± 0.24</td>
<td>-40.086</td>
<td>-9.119</td>
<td>0.895</td>
<td>0.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>150</td>
<td>40</td>
<td>75 ± 28</td>
<td>15.3 ± 7.1</td>
<td>-37.501</td>
<td>-23.694</td>
<td>0.775</td>
<td>0.681</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>200</td>
<td>50</td>
<td>44 ± 20</td>
<td>16.6 ± 7.8</td>
<td>-32.869</td>
<td>-24.402</td>
<td>0.560</td>
<td>0.714</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>100</td>
<td>40</td>
<td>26 ± 11</td>
<td>7.2 ± 3.2</td>
<td>-22.279</td>
<td>-20.086</td>
<td>0.067</td>
<td>0.513</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>150</td>
<td>50</td>
<td>13 ± 4</td>
<td>10.1 ± 3.0</td>
<td>-28.299</td>
<td>-17.147</td>
<td>0.348</td>
<td>0.375</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>200</td>
<td>30</td>
<td>131 ± 29</td>
<td>33.5 ± 14.2</td>
<td>-42.345</td>
<td>-30.501</td>
<td>1.000</td>
<td>1.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>100</td>
<td>50</td>
<td>11 ± 3</td>
<td>5.8 ± 2.1</td>
<td>-20.828</td>
<td>-15.269</td>
<td>0.000</td>
<td>0.288</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>150</td>
<td>30</td>
<td>15 ± 4</td>
<td>12.6 ± 6.2</td>
<td>-23.522</td>
<td>-22.007</td>
<td>0.126</td>
<td>0.602</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>200</td>
<td>40</td>
<td>46 ± 19</td>
<td>8.5 ± 3.3</td>
<td>-33.255</td>
<td>-18.588</td>
<td>0.580</td>
<td>0.442</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Grey coefficient \(\zeta_i(k)\) is measured by the following Equation (3):

\[
\zeta_i(k) = \frac{\Delta_{min} + \psi \Delta_{max}}{x_i + \psi \Delta_{max}}
\]

where \(\Delta_{min}\) is the minimum variation obtained in grey coefficient values, which is therefore 0, and \(\Delta_{max}\) is the maximum one, corresponding to 1. \(\psi\) is a coefficient aimed at equalizing the values of the measured parameters, hence WR and COF, which is therefore equal to 0.5, as described, e.g., in [28].

The grey rate is the average of the two grey coefficients, which are subsequently ranked in decreasing order, as from Table 4. The rank indicates that the best conditions are obtained with experiment n.7.
Table 4. Grey coefficient, grey rate, and rank of the different tests.

<table>
<thead>
<tr>
<th>Load (N)</th>
<th>Speed (rpm)</th>
<th>Sliding Distance (m)</th>
<th>Grey Coefficient WR</th>
<th>Grey Rate COF</th>
<th>Grey Rate Rank</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>100</td>
<td>30</td>
<td>0.358</td>
<td>1.000</td>
<td>0.679</td>
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<tr>
<td>10</td>
<td>150</td>
<td>40</td>
<td>0.392</td>
<td>0.423</td>
<td>0.408</td>
</tr>
<tr>
<td>10</td>
<td>200</td>
<td>50</td>
<td>0.472</td>
<td>0.412</td>
<td>0.442</td>
</tr>
<tr>
<td>20</td>
<td>100</td>
<td>40</td>
<td>0.881</td>
<td>0.493</td>
<td>0.637</td>
</tr>
<tr>
<td>20</td>
<td>150</td>
<td>50</td>
<td>0.590</td>
<td>0.569</td>
<td>0.580</td>
</tr>
<tr>
<td>20</td>
<td>200</td>
<td>30</td>
<td>0.333</td>
<td>0.333</td>
<td>0.333</td>
</tr>
<tr>
<td>30</td>
<td>100</td>
<td>50</td>
<td>1.000</td>
<td>0.634</td>
<td>0.817</td>
</tr>
<tr>
<td>30</td>
<td>150</td>
<td>30</td>
<td>0.799</td>
<td>0.453</td>
<td>0.626</td>
</tr>
<tr>
<td>30</td>
<td>200</td>
<td>40</td>
<td>0.463</td>
<td>0.530</td>
<td>0.496</td>
</tr>
</tbody>
</table>

Further considerations concern the respective importance of the three different characteristics, load, speed, and sliding distance, on the wear performance of the composites. The effects of the grey rational grade are obtained by measuring the average between the three samples that are on the same level of load, speed, and loading distance, as from the sample codes in Table 2. The difference between the top and bottom values of the grey relational grades (Max-Min) was also calculated, and all these data are reported in Table 5.

Table 5. Effects of grey rational grade with different factors and levels (1, 2, and 3).

<table>
<thead>
<tr>
<th>Factors</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>Max-Min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load</td>
<td>0.510</td>
<td>0.465</td>
<td>0.646</td>
<td>0.181</td>
</tr>
<tr>
<td>Speed</td>
<td>0.711</td>
<td>0.538</td>
<td>0.424</td>
<td>0.267</td>
</tr>
<tr>
<td>Sliding distance</td>
<td>0.546</td>
<td>0.514</td>
<td>0.613</td>
<td>0.099</td>
</tr>
</tbody>
</table>

Speed variation measurements reported the greatest difference between the highest and lowest values of the average grey relational grades, followed by load and sliding distance. The three parameters were rated in order of significance to the multi-performance characteristics procedure, with the respective values, revealing clear differences, of 0.267 > 0.181 > 0.099. The grey rational grades are summarized in Figure 3. In practice, these values highlighted the relative importance of the controllable factors to the wear volume of jute/coir composite with ceramic fillers. This method has been successfully applied in tribology studies of conventional glass fiber polymer composites, for example, in [29].

3.2. Performing Statistical Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) is a method for determining which parameter has the most significant impact on assessing the characteristics of the material, in this case wear ones. Fundamentally, the higher the grey grade, the better the overall performance qualities. As a result of grey grade analysis, though with obvious restrictions, due to the limited number of samples tested, the best quality parameter for simultaneously maximizing tribological properties is to keep the speed at level 1, the load at level 3, and sliding distance also at level 3. The best multi-performance features and the highest grey relational grade were provided by this combination. As a consequence, in experiment 7, the suitable settings for the optimum multi-performance features such as wear rate and COF were chosen. The ANOVA was implemented by measuring the sum of squares (SOS) obtained by summing up the squared differences between different levels (L) of the three factors, as per Equation (4). The values obtained are reported in Table 6.

\[
SOS = (L_1 - L_2)^2 + (L_2 - L_3)^2 + (L_3 - L_1)^2
\]  

(4)
Speed variation measurements reported the greatest difference between the highest and lowest values of the average grey relational grades, followed by load and sliding distance. The three parameters were rated in order of significance to the multi-performance characteristics procedure, with the respective values, revealing clear differences, of 0.267 > 0.181 > 0.099. The grey rational grades are summarized in Figure 3. In practice, these values highlighted the relative importance of the controllable factors to the wear volume of jute/coir composite with ceramic fillers. This method has been successfully applied in tribology studies of conventional glass fiber polymer composites, for example, in [29].

Figure 3. Grey rational grades.

Table 6. ANOVA study of grey rational grade.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Degrees of Freedom (DOF)</th>
<th>Sum of Squares (SOS)</th>
<th>Mean</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load</td>
<td>2</td>
<td>0.0531</td>
<td>0.0265</td>
<td>27.8</td>
</tr>
<tr>
<td>Speed</td>
<td>2</td>
<td>0.1221</td>
<td>0.0610</td>
<td>64.1</td>
</tr>
<tr>
<td>Sliding distance</td>
<td>2</td>
<td>0.0153</td>
<td>0.0077</td>
<td>8.1</td>
</tr>
<tr>
<td>Total</td>
<td>8</td>
<td>0.1905</td>
<td>0.0952</td>
<td>100</td>
</tr>
</tbody>
</table>

The results indicate the large predominance of the speed factor over the load one, with a very limited influence of the sliding distance on the obtained results.

3.3. Scanning Electron Microscope (SEM) Analysis

A microstructural investigation was carried out using scanning electron microscope (SEM) images. In this study, SEM images of materials were obtained before wear testing, to verify that the quality of the surface obtained was sufficiently consistent between the different samples examined, and after the wear tests were performed. In particular, three SEM circumstances were investigated, before wear test (sub-images a) and after wear test (sub-images b): high WR with low COF (sample n.1) (Figure 4a,b), high WR with high COF (sample n.6) (Figure 5a,b), and low WR with low COF (optimal sample n.7) (Figure 6a,b). Higher magnification images were provided in the case of sample n.6, in order to explain the coexistence of high coefficient of friction and high wear rate. It needs to be noticed that the initial aspect of the samples can be quite variable, as reported in Figures 4a, 5a and 6a. In particular, some unevenness of the surface is often noticeable due to some imperfections in the resin curing process. Despite this, the characteristics shown by the three samples depicted after wear testing in Figures 4b, 5b and 6b are markedly different. More specifically, on sample n.1 (Figure 4b), the presence of exposed fibers and fillers is indicated, which is a symptom of the resin erosion, though no fragmentation is visible, hence suggesting their behavior is not as brittle as in the case, e.g., of glass fibers [30].
Figure 4. Sample with low COF and high wear rate (sample n.1): (a) Before wear test; (b) After wear test.
When the coefficient of friction is higher, though, as it is the case for sample n.6, ploughing tracks are also clearly visible with long cracks on the surface, which is a typical pattern easily observed on wear tests of jute/polyester composites [31], henceforth suggesting the optimization of parameters, as from this study. As a matter of fact, the situation appears clearly improved in the optimal sample in Figure 6b. Extended comments are given in Section 4.

Figure 5. Sample with high COF and high wear rate (sample n.6): (a) Before wear test; (b) After wear test.
Figure 6. Sample with optimal conditions (sample n. 7): (a) Before wear test; (b) After wear test.

4. Discussion

Comments on the observation by scanning electron microscope led to some considerations, particularly in light of the existing literature. In particular, in sample 1, which showed a low coefficient of friction and a high wear rate, the worn surface reveals tiny scratches and a clear exposure of the fillers, particularly eggshell powder, which is known to have only partially brittle behavior from previous studies carried out on epoxy composites, and therefore might withstand erosion without fracture [32]. In addition, the presence of many pits and debris indicated the occurrence of a continuous loading effect and a gradual degradation, even in the presence of velocities much lower than in other similar studies on the introduction of cellulosic waste in epoxy resin, such as in [33]. On the other hand, this is
expected as previous tribology studies on jute fiber composites have evidenced the limited performance of polyester ones when compared to epoxy ones [34], though unsaturated polyester is particularly compatible with quite highly lignified fibers, such as jute [35] and coir [36].

At greater speeds and loads, such as in sample 6, polyester’s high friction coefficient created significant heat at the interface, softening the resin-rich areas and losing material from the specimen owing to wear. During the testing, it was observed that the polyester debris was produced during sliding and adhered to the disk’s surface and the sample displayed tiny cracks, suggesting fatigue stress. It is likely that the COF and wear rate increased as a result of this situation. On the other hand, the presence of fillers reduced the formation of ploughing surfaces with respect to recent studies on other hybrid natural composites, such as banana/kenaf [37]. It needs to be emphasized that the early appearance of ploughing even for friction at low speeds does represent a limitation of the use of natural fiber composites for tribology applications. This occurs even for high performance natural fibers, such as flax, where the deviations from designed fiber orientation would also make machining more difficult and prone to ploughing effect [38].

In contrast to the issues presented above, sample 7’s SEM picture indicated no flaking or degradation of the fibers. After the wear test, the case revealed a type of compact packing and a smooth surface.

Since the fillers that are self-lubricating are present in all three of the foregoing situations, they would function in ideal conditions as a barrier against additional wear. This might function as a lubricant, preventing wear and weight loss by reducing friction. The brittleness of the surface was reduced by mixing the fillers with the matrix, which prevented the flake wear. This happened with particular evidence in the A3B1C3 circumstance. As a result, the three parameters (load = 30 N, speed = 100 rpm, sliding distance = 50 m) represented an ideal combination for potential use of jute-coir composites with eggshell powder and nanoclay fillers for use in automotive components that required relative motion, such as brushes, washers, and spacers. In this case, the ceramic fillers may compete with a hardening function typically played in natural fiber composites for wear-resistant structures by the introduction of silica [39]. In general terms, introduction of natural fibers, such as, e.g., kenaf [40], jute [41], or coir [42], does improve the wear characteristics of unsaturated polyester resins. However, in a specific case, the higher degree of complication involved in the fabrication of hybrids with two different fibers and other fillers would be compensated in another sense by the possible exploitation of waste or low value items from other very diffuse productive systems (examples can be coconut and sugarcane [43]) in the field of friction-exposed materials. The success of the operation appears so far limited to low speeds, of less than 1 m/s, and loads not exceeding a few tens of Newtons, while long sliding distances do not appear to represent an issue. It can be suggested that a better compatibilization between the fibers and the matrix, allowing easier penetration of the latter, would increase the performance. On the other hand, ceramic fillers, such as eggshell powder and nanoclay, did appear effectively integrated in the resin, at least in amounts not exceeding a few percent, which are sufficient to the hardening purpose.

5. Conclusions

The tribological results of jute/coir fiber reinforced with eggshell powder (ESP)/nanoclay (NC) filler hybrid composites led to the following conclusions:

- Considering speed, applied load, and sliding distance, tribological behavior was influenced most by speed (64.1%), more limitedly by load, and only marginally by sliding distance. Hybrid composites’ friction and wear properties were less affected by the interaction set of parameters than other composite materials.
- Taguchi’s method revealed that the best combination of process parameters for minimizing wear and friction on this composite, was by applying the highest possible sliding distance and load and in contrast the lowest speed.
• By creating a protective barrier between the pin and the counter face, the incorporation of ESP and NC particles improved the wear resistance of hybrid composites. This had a substantial impact on the friction and the wear depth.

• According to the microstructure analysis of worn surfaces, abrasive wear mechanisms primarily occurred on the wear tracks, with occasional indications of adhesive wear mechanisms.

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