Abstract

The tensile, impact and flexural properties of oriented and randomly oriented isora fibre reinforced epoxy composites with special reference to the effect of fibre loading are described in the chapter. The effect of fibre surface modifications like alkali, triton and detergent treatments and ultrasonication, on the properties of the composite is also discussed. Chemical modification of fibre was found to have enhanced the fibre/matrix interactions. The improved fibre/matrix interaction is evident from the improved tensile, flexural and impact properties of the composites. The tensile fractographs of the composites were studied by SEM to analyse the fibre/matrix interaction. The experimental tensile strength values were compared with the theoretical values.

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7.1. Introduction

Epoxy resins find wide applications in the production of composite materials as matrix, in spite of the relatively high cost, due to their easy processability, excellent wetting properties with reinforcements, good weathering resistance, excellent dimensional stability and the wide variety of grades available [1]. Other characteristic features of epoxy resins are their high chemical and corrosion resistance, good thermal and mechanical properties, outstanding adhesion to various substrates, flexibility, low cure shrinkage and good electrical properties [2]. The major advantage of these composite structures over traditional metallic materials like steel and aluminium are their favourable mechanical and physico-chemical properties and high strength to weight ratio. Rong et al. [3] studied the effect of fibre treatment on the mechanical properties of unidirectional sisal-reinforced epoxy composites and concluded that sisal fibres can be effectively modified by chemical and physical treatments. They also reported that adhesion at the interface between sisal bundles and matrix and that between ultimate cells play a key role in determining the mechanical behaviour of the laminates. Van de Weyenberg et al. [4] reported on improving the properties of unidirectional flax fibre reinforced epoxy composites by applying an alkaline fibre treatment and they found that this treatment resulted in a significant increase in both longitudinal and transverse composite properties, due to improvement of the fibre-matrix adhesion. Gassan and Bledzki [5] optimized the mechanical properties of tossa jute fibres using NaOH-treatment process with different alkali concentrations and shrinkages. Shrinkage of the fibres during alkali treatment had the most significant effect on the fibre structure and as a result, on the mechanical properties. Bledzki et al. [6] also studied the influence of fibre treatments on unidirectional hemp and flax fibres reinforced epoxy and polypropylene composites. They have reported that the structural conversion of the fibres and the mechanical properties can be affected in a broad range by using appropriate mercerization parameters like shrinkage,
Isora fibre reinforced epoxy composites

fibre stress, NaOH concentration, temperature and duration of alkali treatment. Bisanda and Ansell [7] reported that silane treatment improved moisture resistance and compressive strength of sisal-epoxy composites. Van de Weyenberg et al. [8] have studied the influence of processing and chemical treatment of flax fibres on the mechanical properties of the fibre reinforced epoxy composites. They inferred that retting action, which removed pectins, seems to be an important step to obtain better quality composites and a combined treatment of alkali and dilute epoxy gives best mechanical properties of the composite. Hepworth et al. [9] described a treatment that enables epoxy resin to penetrate into the cell walls of plant fibres. The treatment involves swelling the plant cell walls with urea solution, washing out the excess urea and then replacing water with alcohol in a graded series. Using this method they were able to make unidirectional flax-epoxy composites that showed large increase in stiffness when compared to composites made from untreated fibre. Gassan and Gutowski [10] reported on the effects of corona discharge and UV treatment on the properties of jute-epoxy composites. They concluded that, to improve the overall mechanical properties of jute/epoxy composites, an appropriate balance need to be achieved between increased polarity of fibre surface and the decrease of fibre strength subsequent to excessive surface oxidation by corona discharge or UV radiation. Jindal [11] studied the impact properties of bamboo fibre reinforced epoxy composites and found that impact strength of these composites is poor. Use of Kenaf fibre as a reinforcing agent in epoxy system was reported by Zimmerman and Losure [12]. J. George et al. [13] investigated mechanical properties of flax fibre reinforced epoxy composites and found that the thermal stability of the fibre is increased after chemical treatment. The mechanical properties of the composites were improved significantly after treatment.

Several other works have been reported using epoxy resin as matrix [14-25].

A detailed study on the tensile, impact and flexural properties of oriented and randomly oriented isora fibre reinforced epoxy composites is proposed in
this chapter. Fibre surface morphology and fibre-matrix adhesion will be analysed by Scanning Electron Microscopy (SEM).

7.2. Results and discussion

7.2.1. Randomly oriented isora fibre reinforced epoxy composites

7.2.1.1. Tensile properties

a) Untreated fibre composite

The effect of fibre loading on the tensile properties of untreated randomly oriented isora-epoxy composite with standard deviation is given in Table 7.1. The variation of tensile strength and Young’s modulus of the composite with fibre loading is shown in Figure 7.1. At low fibre loading, fibres act as flaws and the volume percent of the fibre is not enough to exceed the strength of the matrix [26].

<table>
<thead>
<tr>
<th>Fibre loading (% v/v)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s modulus (MPa)</th>
<th>Elongation at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (neat resin)</td>
<td>45 ± 2.2</td>
<td>1500 ± 60</td>
<td>4.5 ± 0.18</td>
</tr>
<tr>
<td>10</td>
<td>31 ± 1.4</td>
<td>1362 ± 57</td>
<td>2.5 ± 0.13</td>
</tr>
<tr>
<td>24</td>
<td>37 ± 1.6</td>
<td>2180 ± 85</td>
<td>2.5 ± 0.14</td>
</tr>
<tr>
<td>29</td>
<td>46 ± 2.1</td>
<td>2324 ± 88</td>
<td>2.6 ± 0.14</td>
</tr>
<tr>
<td>37</td>
<td>55 ± 2.6</td>
<td>2672 ± 95</td>
<td>2.7 ± 0.15</td>
</tr>
<tr>
<td>45</td>
<td>60 ± 2.8</td>
<td>2784 ± 99</td>
<td>2.8 ± 0.16</td>
</tr>
<tr>
<td>52</td>
<td>56 ± 2.7</td>
<td>2690 ± 97</td>
<td>2.6 ± 0.14</td>
</tr>
</tbody>
</table>

The tensile strength of the composite decreased up to a fibre loading of 24 vol. % and exceeds that of resin only at a loading of 29 vol. %. After that a linear increase is observed. The tensile strength of the composite reached a maximum value at a fibre loading 45 vol. %. The increase in tensile strength compared to neat
resin is 33% at this fibre loading. Thereafter a decrease in tensile strength is observed.

But Young's modulus values showed a linear increase after the initial decrease at 10% fibre loading and reached a maximum value at 45% fibre loading. After that the property decreased for higher fibre loading. Compared to neat resin, this increase in Young's modulus is 85.6%. So the optimum fibre loading is 45%, which is adequate for proper wetting of fibres with epoxy resin. The decrease in tensile properties of the composites with further increase in fibre loading may be due to improper wetting and adhesion between the fibre and resin resulting in inefficient stress transfer. Elongation at break (EB) values of the composites is lower than that of neat resin, indicating improved stiffness. EB value is maximum for the composite containing 45% fibre.

b) Alkali treated fibre composite

The effect of alkali treatment on the tensile strength and Young's modulus of randomly oriented isora-epoxy composite is given in Figures 7.2 (a) and (b) respectively.
Alkali treatment of the fibre improved the tensile properties of the composite. For a fibre loading of 37 vol. %, tensile strength improved by 23.6% and Young's modulus by 19.5%. Elongation at break of the composite decreased on alkali treatment indicating enhanced stiffness of the composite.

The improvement in tensile properties can be attributed to the improved wetting of alkali treated fibre with epoxy resin [27]. On alkali treatment, lignin and hemi cellulose, the cementing materials present in the fibre get dissolved. This makes the interfibrillar region less dense and less rigid as a result of which the fibrils become more capable of orienting themselves along the tensile deformation [28].

The improved wetting of the fibre with the matrix is evident from the SEM photograph of the tensile fracture surface of the alkali treated fibre composite (Figure 7.4) compared to that of untreated fibre composite (Figure 7.3)
7.2.1.2. Flexural properties

a) Untreated fibre composite

The effect of fibre loading on the flexural properties of randomly oriented isora-epoxy composite with standard deviation is given in the Table 7.2. The variation of flexural strength and flexural modulus of the composite with fibre loading is given in Figure 7.5. The flexural strength of the composites containing 10 and 25 vol. % fibres has lower flexural strength compared to neat resin. After that flexural strength of the composite increased with fibre loading and reached maximum at 45% fibre loading, the increase being 19.5%. Flexural strength of the composite decreased at higher fibre loadings.
Table 7.2. Effect of fibre loading on the flexural properties of randomly oriented isora-epoxy composites (fibre length = 30mm)

<table>
<thead>
<tr>
<th>Fibre loading (% v/v)</th>
<th>Flexural strength (MPa)</th>
<th>Flexural modulus (GPa)</th>
<th>Maximum strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (neat resin)</td>
<td>87 ± 3.5</td>
<td>2.32 ± 0.09</td>
<td>4.31 ± 0.18</td>
</tr>
<tr>
<td>10</td>
<td>65 ± 2.7</td>
<td>2.66 ± 0.11</td>
<td>2.09 ± 0.08</td>
</tr>
<tr>
<td>25</td>
<td>83 ± 3.7</td>
<td>4.61 ± 0.19</td>
<td>2.65 ± 0.12</td>
</tr>
<tr>
<td>37</td>
<td>91 ± 4.0</td>
<td>5.90 ± 0.25</td>
<td>2.37 ± 0.10</td>
</tr>
<tr>
<td>45</td>
<td>104 ± 4.3</td>
<td>6.12 ± 0.26</td>
<td>2.65 ± 0.13</td>
</tr>
<tr>
<td>52</td>
<td>96 ± 4.4</td>
<td>5.95 ± 0.24</td>
<td>2.52 ± 0.12</td>
</tr>
</tbody>
</table>

Flexural modulus of the composite increased linearly right from the beginning and reached maximum at 45% fibre loading, the increase being 163%, after which a lowering of modulus was observed. So the optimum fibre loading for randomly oriented isora-epoxy composite is 45 vol. %, which is adequate for proper wetting of the fibre and thereby maximum stress transfer between the fibre and resin. The lower flexural strength of the composite compared to neat resin at lower fibre loadings is due to inefficient stress transfer. Increase in flexural strength with increasing fibre content is due to increased interaction and adhesion between fibre and resin. The decrease of flexural strength at higher fibre loading is due to increased fibre-to-fibre interaction and dispersion problems.

Figure 7.5. Variation of flexural strength and flexural modulus with fibre loading, of randomly oriented isora-epoxy composite
b) **Alkali treated fibre composite**

On alkali treatment, flexural properties of the composite were found to be improved. At a fibre loading of 37 vol. %, the flexural strength of the composite improved by 9% and a marginal increase of 3.5% in flexural modulus was also observed (Figure 7.6). The increase in flexural strength and flexural modulus on alkali treatment of fibre can be attributed to the formation of rough fibre surface leading to the improved wetting of fibres with the matrix.

![Figure 7.6. Effect of alkali treatment on the flexural strength and flexural modulus of randomly oriented isora-epoxy composite.](image)

7.2.1.3. Impact properties

a) **Effect of fibre loading**

The variation of impact strength of untreated randomly oriented isora-epoxy composites with fibre loading is given in Figure 7.7. The impact strength of the composites is increasing linearly with fibre loading up to 56%. The increase in impact strength is not significant above 56% fibre loading. Compared to neat resin (67 J/m), there is 134% increase in impact strength for the composite containing 56% fibre by volume.

The improvement in impact strength of the composite may be attributed to the extra energy needed for the fibre pull out, debonding or redistribution of stress.
b) Effect of alkali treatment

The effect of alkali treatment on the impact strength of randomly oriented isora-epoxy composite containing 34 vol. % fibre is given in Figure 7.8.
Alkali treatment of fibre lowered the impact strength of the composite by 30% compared to untreated fibre composite. This lowering of impact strength of the alkali treated fibre composite may be attributed to the stronger interfacial bonding between the fibre and the resin. A weak interface will not facilitate efficient stress transfer resulting in a weak but tough composite. But the composites having strong interface will have low toughness value compared to the one having weaker interface. The low impact strength of the alkali treated fibre composite is assumed to be due to the strong interface as evidenced from the mechanical properties of the composite.

7.2.2. Oriented long isora fibre reinforced epoxy composites
7.2.2.1. Tensile properties

a) Effect of fibre loading on untreated fibre composite

The effect of fibre loading on the tensile properties of the untreated, oriented long isora fibre-epoxy composite with standard deviation are given in the Table 7.3. The variation of tensile strength and Young’s modulus of the composite with fibre loading is shown in the Figure 7.9.

<table>
<thead>
<tr>
<th>Fibre loading (% v/v)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s modulus (GPa)</th>
<th>Elongation at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (neat resin)</td>
<td>45 ± 2.2</td>
<td>1.50 ± 0.06</td>
<td>4.46 ± 0.18</td>
</tr>
<tr>
<td>31</td>
<td>109 ± 4.3</td>
<td>3.8 ± 0.17</td>
<td>3.26 ± 0.13</td>
</tr>
<tr>
<td>36</td>
<td>151 ± 6.3</td>
<td>5.15 ± 0.23</td>
<td>3.41 ± 0.14</td>
</tr>
<tr>
<td>40</td>
<td>178 ± 7.8</td>
<td>5.99 ± 0.28</td>
<td>3.56 ± 0.16</td>
</tr>
<tr>
<td>49</td>
<td>200 ± 8.0</td>
<td>6.55 ± 0.30</td>
<td>4.42 ± 0.20</td>
</tr>
<tr>
<td>53</td>
<td>198 ± 7.9</td>
<td>6.48 ± 0.29</td>
<td>4.07 ± 0.18</td>
</tr>
</tbody>
</table>

Tensile strength and modulus of the composite increased regularly with fibre loading and reached maximum at 49% loading. The increase in tensile strength is
344% and increase in Young’s modulus is 337%, compared to neat epoxy resin. The increase in tensile strength and modulus are not significant at higher fibre loadings.

**Figure 7.9.** Variation of tensile strength and Young’s modulus with fibre loading of untreated oriented long isora-epoxy composites.

So the optimum fibre loading for the tensile properties of the composite is 49% by volume at which there is maximum wetting of the fibre and effective stress transfer at the fibre/matrix interface. The decrease in tensile properties at higher fibre loadings may be due to improper wetting and adhesion. EB values of the composite are lower than that of neat resin and increase regularly with fibre loading, reached maximum at the optimum fibre loading of 49 vol. %.

**b) Effect of fibre treatments**

The effect of various surface modifications of the fibre on the tensile properties of the oriented long isora fibre-epoxy composites, with standard deviation is given in the Figures 7.10, 7.11and 7.12. The effect of fibre treatment on the tensile strength of the composites is given in Figure 7.10. All fibre treatments resulted in improvement in tensile properties. The tensile strength increased in the order UT < US < DT < AT < TT. The percentage increase in tensile strength of the treated fibre composites compared to untreated fibre composite is as follows: TT-10, AT-5.5, DT-5 and US-2.5.
Figure 7.10. Effect of various fibre treatments on the tensile strength of oriented long isora-epoxy composites

Figure 7.8 shows the effect of fibre treatment on the Young's modulus of the composites. The increase in Young's modulus values of the composites on surface modification of the fibre follows the same trend as in the case of tensile strength. The percentage increase in the property compared to UT fibre composite is: TT-26.9, AT-12.3, DT-10.5 and US-9. It can be observed that fibre treatments have more effect on stiffness of the composite than on tensile strength.

Figure 7.11. Effect of various fibre treatments on Young's modulus of oriented long isora-epoxy composites
The effect of fibre treatment on % elongation at break of the composite is given in Figure 7.12. The EB values of the treated fibre composites are lower than that of untreated fibre composite, supported by the high modulus values of the treated fibre composites.

![Graph showing effect of various fibre treatments on % elongation at break of oriented long isora-epoxy composites](image)

**Figure 7.12. Effect of various fibre treatments on % elongation at break of oriented long isora-epoxy composites**

The improvement in tensile properties can be attributed to the improved adhesion between the fibre and the resin after chemical treatment of fibre. Fibre treatment washes away the surface impurities and develops micro porosity with many pits and holes on the fibre surface by the removal of globular protrusions of fibre surface present in the untreated fibre. This leads to a larger area of contact and greater mechanical interlocking between fibre and matrix, making the interfacial adhesion stronger and the mechanical properties higher. The decrease in tensile strength and Young’s modulus at higher fibre loadings is due to improper wetting and poor adhesion between the fibre and matrix resin.

The Scanning electron photographs of the tensile fracture surface of oriented long isora-epoxy composites are given in the Figures 7.13 (a) to (d)
Figure 7.13. (a)-(d) Tensile fracture surface of treated, oriented long isora-epoxy composites at magnifications $\times 200$ and $\times 700$.
Fibre pull out and debonding are clear in the SEM photograph of untreated fibre composite whereas improved fibre-matrix adhesion is evident in the SEM photographs of various treated fibre composites.

### 7.2.2.2. Flexural properties

#### a) Untreated fibre composite

The effect of fibre loading on the flexural properties of untreated fibre composites, with standard deviation is given in Table 7.4. The variation of flexural strength and flexural modulus of the composite are shown in Figure 7.14. The flexural strength and flexural modulus of the composites are higher than that of neat resin. The flexural strength and flexural modulus of the composite increased regularly with fibre loading, reached maximum at a fibre loading of 45% by volume, then decreased for higher loadings.

**Table 7.4. Effect of fibre loading on the flexural properties of untreated oriented long isora-epoxy composites**

<table>
<thead>
<tr>
<th>Fibre loading (%)</th>
<th>Flexural strength (MPa)</th>
<th>Flexural modulus (GPa)</th>
<th>Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (neat resin)</td>
<td>87 ± 3.5</td>
<td>2.32 ± 0.09</td>
<td>4.31 ± 0.18</td>
</tr>
<tr>
<td>33</td>
<td>162 ± 6.8</td>
<td>7.85 ± 0.34</td>
<td>3.11 ± 0.13</td>
</tr>
<tr>
<td>37</td>
<td>180 ± 7.9</td>
<td>10.3 ± 0.45</td>
<td>3.20 ± 0.14</td>
</tr>
<tr>
<td>41</td>
<td>195.5 ± 8.4</td>
<td>11.62 ± 0.53</td>
<td>3.29 ± 0.15</td>
</tr>
<tr>
<td>45</td>
<td>200 ± 9</td>
<td>12.64 ± 0.57</td>
<td>3.39 ± 0.17</td>
</tr>
<tr>
<td>50</td>
<td>190 ± 8.3</td>
<td>11.42 ± 0.50</td>
<td>3.17 ± 0.13</td>
</tr>
</tbody>
</table>

Compared to neat resin, the increase in flexural strength of the composite is 86% at a fibre loading of 33% and 131% at 45% fibre loading. The corresponding increase in flexural modulus is 238% at 33% fibre loading and 453%, at 45% fibre loading. So the optimum fibre loading for flexural properties of the untreated fibre composite is 45% by volume. Increase in flexural strength with increasing fibre loading is due to increased interaction
between fibre and matrix whereas the decrease of flexural strength at higher fibre loading is due to increased fibre-to-fibre interaction and dispersion problems.

![Graph showing flexural strength and modulus vs. fibre loading](image)

**Figure 7.14.** Effect of fibre loading on the flexural strength and flexural modulus of untreated oriented long isora-epoxy composites

**b) Effect of fibre treatments**

The effect of various surface modifications of the fibre on the flexural properties of the oriented long isora fibre-epoxy composites, with standard deviation is given in the Figures 7.15, 7.16 and 7.17. Figure 7.15 shows the effect of fibre treatment on the flexural strength of the composites compared to untreated fibre composite. Flexural properties of the composite are found to be improved by all fibre treatments. The flexural strength increased in the order UT < AT < DT < TT < US. The percentage increase in flexural strength of the treated fibre composites compared to UT fibre composite is as follows: AT-38, DT-41.5, TT-50.3 and US-59.
The effect of fibre treatment on the flexural modulus of the oriented long isora-epoxy composites, with standard deviation is given in the Figure 7.16. Flexural modulus of the composite varied in the same manner as that of flexural strength, on fibre treatment. Compared to untreated fibre composite the percentage increase in flexural modulus of the treated fibre composite is: AT-35, DT-36.4, TT-70.7 and US-80.
On fibre treatment, flexural strain of the composite varied in the order: TT < US < DT < AT < UT. (Figure 7.17). The decrease in percentage strain of the treated fibre composites compared to the untreated fibre composite is TT-42.4, US-37.5, DT-35.9 and AT-7.7.

![Diagram showing flexural strain for TT, US, DT, AT, UT treatments](image)

**Figure 7.17.** Effect of various fibre treatments on the flexural strain of oriented long isora-epoxy composites

### 7.2.2.3. Impact properties

#### a) Untreated fibre composite

Figure 7.18 shows the variation of impact strength of the untreated oriented long isora-epoxy composites with fibre loading. Impact strength increased with fibre loading, reached a maximum value at 55% by volume of fibre. Compared to neat resin, the increase in impact strength is 166%. With further increase in fibre loading, there is no significant increase in impact strength. The fibres play an important role in the impact resistance of the composites as they interact with the crack formation in the matrix and act as stress transferring medium.
b) Treated fibre composite

The effect of various fibre modifications on the impact strength of oriented long isora-epoxy composite is given in the Figure 7.19. On fibre treatment, impact strength of the composites improved, the improvement followed the order: TT > AT > US > DT > UT. Compared to untreated fibre composite the percentage increase in impact strength is: DT-29.6, US-32.5, AT-33.7 and TT-36.2.
The impact failure of a composite occurs by factors like matrix fracture, fibre/matrix debonding and fibre pull out. Fibre pull out is found to be an important energy dissipation mechanism in fibre reinforced composites [29].

![Optical photograph of the impact fracture surface of oriented isora-epoxy composite](image)

**Figure 7.20.** Optical photograph of the impact fracture surface of oriented isora-epoxy composite

The applied load transferred by shear to fibres may exceed the fibre/matrix interfacial bond strength and then debonding occurs. When the stress level exceeds the fibre strength, fibre fracture occurs. The fractured fibres may be pulled out of the matrix, which involves energy dissipation [30]. The impact fracture of the composites was brittle as evident from Figure 7.20 and not hinge type like their polyester counterpart (Figure 6.12). This indicates stronger interfacial adhesion in the epoxy composite. This is supported by the lower impact strength of oriented isora-epoxy composites compared to oriented isora-polyester composites (compare Figures 6.10 and 7.18). Fibre breakage is the main energy dissipating mechanism here.

### 7.3. Theoretical modeling

#### 7.3.1. Randomly oriented isora-epoxy composites

The theoretical values of tensile strength calculated using Series and Hirsch model are found to be higher than the experimental tensile strengths of untreated randomly oriented isora-epoxy composites, shown in Figure 7.21. But the
theoretical and experimental Young's modulus values are in good agreement which is evident in Figure 7.22, indicating effective stress transfer between isora fibre and epoxy resin.

Figure 7.21. Comparison of experimental and theoretical tensile strengths of untreated randomly oriented isora-epoxy composites

Figure 7.22. Comparison of experimental and theoretical Young's modulus of untreated randomly oriented isora-epoxy composites
7.3.2. Oriented isora-epoxy composites

The theoretical and experimental values of tensile strength and Young’s modulus of the composite are plotted in Figures 7.23 and 7.24 respectively. In both cases, there is better agreement between Hirsch model values and the experimental ones whereas the ROM values are higher than experimental values.

**Figure 7.23.** Comparison of experimental and theoretical tensile strengths of untreated oriented long isora-epoxy composites

**Figure 7.24.** Comparison of experimental and theoretical Young’s modulus of untreated oriented long isora-epoxy composites
7.4. Conclusion

Randomly oriented untreated isora-epoxy composites showed maximum properties at 45% fibre loading. Alkali treatment of fibre improved tensile and flexural properties of the composite but decreased the impact strength.

The tensile properties of oriented long isora-epoxy composites were maximum at 49% fibre loading and the flexural properties at 45% fibre loading. All fibre treatments resulted in enhancement of properties. Triton treatment showed maximum improvement in tensile and flexural properties. The tensile fractographs of the composites were studied by SEM and it further showed improved fibre-matrix interaction for the samples which showed high strength and moduli.

Impact strength of the composite was maximum at 55% fibre loading. Fibre treatments resulted in improved impact strength, which was maximum for triton treatment.

References


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