CHARACTERIZATION AND COMPARISON OF TREATED AND UNTREATED RICH HUSH ASH & FLY ASH FOR METAL MATRIX COMPOSITES

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Abstract:
Rice Husk ash and fly ash are agricultural and coal wastes respectively. These are produced in abundance globally and poses risk to health as well as environment. Thus their effective, conducive and eco-friendly utilization has always been a challenge for scientific community. The fly ash has been used as reinforcement for improved mechanical properties of composites (1,3-5,9). Rice husk ash can also be used for similar applications as its composition is almost similar to that of fly ash. This paper mainly deals with identification of characteristics of both the fly ash and rice husk ash using spectroscopic and microscopic analysis. SEM, XRD, XRF and FTIR spectroscopic methods were used for the characterization of treated and untreated ashes. The results were compared and it was observed that both ashes possesses nearly same chemical phases and other functional groups thus proposing the use of rice husk ash as reinforcement like fly ash in Metal Matrix Composites (MMCs) specifically for wear resistance applications.

Keywords - Rice Husk Ash, Fly ash, SEM, XRD, FTIR, MMC

1. Introduction

Metal matrix composites (MMCs) are the most versatile and outstanding hybrid composites possessing the combined benefits of metals and ceramics, resulting in yield of widespread, extensive and multiple applications. MMCs enhance physical and mechanical properties of the products in which these are embedded or implanted. Among the various types of MMCs, particulate-reinforced composites are the simplest and widely used because of their economical nature [6,8,9,11]. Fly ash (FA) - a particulate-reinforcement, consists of a potential discontinuous dispersoids used in metals to form composites. Since fly ash is a low-cost and less-dense reinforcement available in large quantities as a waste by-product in thermal power plants [8,10,3] its usage is highly prevalent in metal matrix composites. There are two types of fly ash namely, precipitator (solid particle) and cenosphere (hollow particle) [6, 8,9]. The major chemical constituents of fly ash are SiO₂, Al₂O₃, Fe₂O₃ and CaO. Mineralogically, fly ash constitutes the alumino-silicate glasses containing quartz, mullite, hematite, magnetite, ferrite, spinel, anhydride and alumina [8,11].

Rice husk ash (RHA) is an agricultural waste and by-product of rice husk. It was estimated to be produced globally 21,000,000 tonnes in 2002 [7] and is a major causative seasonal environmental and health threat. The major chemical constituents of rice husk ash are SiO₂, Al₂O₃, Fe₂O₃ and CaO [7,2,12,4]. Rice husk ash is one of the most silica rich raw materials containing about 90 – 98% silica after complete combustion among the family of other agro wastes [7]. As the fly ash and rice husk ash possesses almost similar chemical composition, it has been proposed by the investigators to use RHA as reinforcement in MMCs. Surface treatment of FA and RHA particles is a prerequisite for getting an acceptable level of its dispersion in metals and alloys with minimum agglomeration and porosity [8,1]. The present paper thus mainly deals with characterization and comparison of chemical, physical and morphological properties of RHA (100 µm) and FA (100 µm) to be used in MMC.
2. Method and Materials

2.1 Method

Sedimentation process was carried out on the mixture of fly ash (500g) and rice husk ash (500g) each with double deionised water (1000 ml) to remove the soluble inorganic matter in order to eliminate pores clogging. Both the ashes were then heated at 200°C for 2 hours in muffle furnace to remove the water particles and allowed to cool in furnace. Ball milling was carried out on both the ashes for 6 hours at 150 rpm to separate agglomerated particles of ashes and to further decrease their particle size. Both the ashes were divided into two parts each. One part of each ash was meant for further chemical treatment and other part remained untreated. Each sample of both ashes were mixed with 1M HCl solution in the ratio of 1:2 (Ash : HCl), filtered and further heat treated at 200°C for 2 hours in closed muffle furnace and allowed to cool in furnace. Again ball milling was carried out at 150 rpm for 6 hours to remove agglomeration and to make fine ash powders. After ball milling, both the ashes were stored in desiccators separately.

2.2 Materials

Rice husk ash was acquired from Amrit Banaspati Company Ltd, Punjab, India and Fly ash was procured from Abhishek Industries, Punjab, India. Chemical compositions of as-received rice husk ash and fly ash are illustrated in Table 1 and Table 2 respectively.

Table 1: Chemical Composition of as-received Rice Husk Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>4.21</td>
<td>0.2</td>
<td>0.40</td>
<td>2.54</td>
<td>0.30</td>
<td>0.07</td>
<td>92</td>
</tr>
</tbody>
</table>

Table 2: Chemical Composition of as-received Fly Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>BaO</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>31.98</td>
<td>0.16</td>
<td>0.70</td>
<td>3.55</td>
<td>1.03</td>
<td>0.44</td>
<td>0.07</td>
<td>61.07</td>
</tr>
</tbody>
</table>

Both the ashes were cleaned with double deionised water and then activated with 1 M HCl solution.

2.3 Chemical Characterization

Chemical composition of untreated RHA and FA after sedimentation process and ball milling was evaluated and major oxides were determined using X-Ray Fluorescence spectroscopy and the results thus obtained are depicted in Tables 3-4.

Table 3: Chemical Composition of untreated Rice Husk Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>4.10</td>
<td>0.34</td>
<td>0.64</td>
<td>2.15</td>
<td>0.64</td>
<td>0.15</td>
<td>91.98</td>
</tr>
</tbody>
</table>

Table 4: Chemical Composition of untreated Fly Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>BaO</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>29.62</td>
<td>0.13</td>
<td>0.72</td>
<td>3.53</td>
<td>0.99</td>
<td>0.34</td>
<td>0.10</td>
<td>64.57</td>
</tr>
</tbody>
</table>

2.4 Mineralogical Characterization

Both untreated and treated samples of RHA and FA were evaluated for their mineralogical characteristics by employing X-Ray Diffraction (XRD) spectrometer (Panalytical X’ Pert Pro) studies using Cu K-α radiations with nickel metal as beta filter. Each sample was scanned from 2θ ranging from 5-60°. Presence of minerals was confirmed with the help of data files presented by Joint Committee on Powder Diffraction Standard (JCPDS). Diffraction patterns of untreated and treated rice husk ash and fly ash are presented in Figures 1– 4.
2.5 Scanning Electron Microscopy (SEM)
Morphology of untreated and treated RHA and FA were studied using Scanning Electron Microscopy (JSM 6100, JEOL). Various samples were placed on sample holder and the images were captured under various magnifications. Prior to it, samples were applied with the gold coating to avoid charge effect, so to obtain clear images. SEM micrographs of untreated and treated RHA and FA are demonstrated in Figures 5-8.
2.6 Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups present in untreated and treated RHA and FA were confirmed using Fourier transform infrared spectroscopy (Perkin Elmer-Spectrum RX-IFTIR, India). It has resolution of 1 cm\(^{-1}\) and scan range of 4000 cm\(^{-1}\) to 250 cm\(^{-1}\). Ash samples were scanned from 4000 cm\(^{-1}\) to 500 cm\(^{-1}\) and results are exhibited in Figures 9-14.

![Figure 9: FTIR of Untreated Rice Husk Ash](image)

![Figure 10: FTIR of Treated Rice Husk Ash](image)

![Figure 11: Comparison of Untreated and Treated Rice Husk Ash](image)
3. Results and Discussion

3.1 X-Ray Fluorescence (XRF) Results
Chemical compositions of untreated and treated samples of rice husk ash and fly ash are depicted in Table 3-6 respectively.

Table 5: Chemical Composition of treated Rice Husk Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>3.06</td>
<td>0.56</td>
<td>0.15</td>
<td>2.67</td>
<td>0.73</td>
<td>0.36</td>
<td>91.57</td>
</tr>
</tbody>
</table>

Table 6: Chemical Composition of treated Fly Ash

<table>
<thead>
<tr>
<th>Element</th>
<th>Al₂O₃</th>
<th>BaO</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>MgO</th>
<th>Na₂O</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>25.62</td>
<td>0.09</td>
<td>0.69</td>
<td>3.14</td>
<td>0.78</td>
<td>0.56</td>
<td>0.24</td>
<td>69.45</td>
</tr>
</tbody>
</table>

Comparison of chemical composition of both as-received and untreated RHA and FA as represented in Tables 1 - 4 shows the mild change in the chemical composition of both the ashes as dust particles get washed out on treatment. The heated and chemically treated ashes with the HCl solution increase the specific surface area and induce changes in its surface properties [1]. Further chemical analysis of both untreated and treated RHA and FA as shown in Tables 3-6 confirmed their chemical similarity. SiO₂, Al₂O₃ and Fe₂O₃ were found to be major constituents of both ashes. Silicon dioxide and alumina are known to be among the hardest substances. Some other oxides viz. CaO, MgO, K₂O, Na₂O and BaO were also found to be present in traces. The presence of hard elements like SiO₂, Al₂O₃ and Fe₂O₃ suggested the researcher to use RHA as particulate reinforcement in various matrices. Therefore in light of similar chemical composition of RHA and FA, the present work suggests to use RHA as particulate in metal matrix composites.
3.2 X-Ray Diffraction (XRD) Analysis

Phase analysis and nature (crystalline/amorphous) of both ashes was confirmed using XRD technique. Diffraction pattern of untreated and treated RHA confirmed the presence of same major phases in both powder samples. Silicon (Si) was observed to be the major phase and remained unchanged even after chemical treatment. According to JCPDS card of Si, its presence was confirmed at 26.7460° (2θ) angle. In diffractograms shown in Figure 1 and 3 of untreated and treated RHA, highest peak of Si was observed at same angle 26.7460°, similar to information given by JCPDS standard.

Diffractograms of untreated and treated FA powder were also found to remain unchanged as shown in Figure 2 and 4. Si was noted to be the only major constituent as shown by highest peak located at an angle of 26.7460°. Some other elements can also be observed, but in traces in the form of small peaks.

RHA powder was observed to be amorphous as seen in Figure 1 and 3 due to agglomerated peaks. Heating of powder at 200°C for 2 hours after water treatment followed by again heating at 200°C for 2 hours might not evaporate water completely caused RHA to be of amorphous nature. On the other hand, FA was observed to be of crystalline nature due to well defined and segregated peaks as shown in Figure 2 and 4.

3.3 Scanning Electron Microscopy (SEM) Analysis

Particle Morphology of untreated and treated RHA can be seen in back scattered electron (BSE) micrographs as shown in Figures 5 and 6 at magnifications of 430X and 220X respectively. RHA particles were observed to be of solid in nature (precipitators), but irregular in size. Some spherical shape particles can also be seen in these micrographs. In Figure 5, particles can be seen as agglomerated and sticked to each other which may be due to presence of water.

Particle morphology of untreated and treated FA can be observed in Figures 7–8 at different magnifications. FA particles were observed to be irregular in shape, size and solid in cross section.

3.4 Fourier Transform Infrared (FTIR) Analysis

Mainly four peaks were detected in all cases of untreated and treated rice husk ash and fly ash as visible in Figure 9 to 14. These are located at 3465, 1653.2, 1094, 797 and 465.2 cm⁻¹. Criado et al. [3] has shown that the presence of quartz in the original ash gives rise in the IR spectrum to a series of bands located at 1150, 1084, 796–778, 697, 668, 522 and 460 cm⁻¹. The presence of mullite, in turn, is responsible for a series of bands at around 1180–1130 cm⁻¹ and 560–550 cm⁻¹. Quartz, mullite and the vitreous phase of the ash overlap in the area between 1200 cm⁻¹ and 900 cm⁻¹.

Hence Quartz, Mullite and vitreous phases are confirmed to be present. More over the peaks in treated and untreated cases does not show any variations. Since all the peaks are near about at same values, which shows that the functional groups present in both the powders are nearly same.

4. Conclusion

1) XRF and XRD studies revealed the presence of almost similar elements in both untreated and treated FA and RHA powders. Hardest substances like SiO₂, Al₂O₃ were present as major constituents which can be used as particulate reinforcements in composites for wear-resistant applications.

2) FTIR graphs showed that Quartz, Mullite and Vitreous phases were present in both powder and proposed to use RHA as particulate reinforcement in MMCs.

5. References