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SYNTHESIS, CHARACTERIZATION AND HARDNESS STUDIES OF NANO RICE HUSK ASH REINFORCED AL6061 NANOCOMPOSITES

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Abstract

Rice husk ash is produced after combustion of rice husk, which is an industrial waste. Rice husk ash consists of 92% of SiO_2. This paper describes the synthesis of completely combusted white coloured Nano Rice Husk Ash to nanoparticle size using planetary ball mill, characterization of Rice Husk Ash and Nano Rice Husk Ash using energy dispersive X-ray Analysis, X-ray Diffraction, Dynamic Light Scattering, Scanning Electron Microscopy, Thermogravimetric Analysis and Differential Thermal Analysis. For synthesizing nanoparticles, the macro size white coloured Rice Husk Ash is taken in a planetary ball mill and milled until getting nano-size particles. The characterization results revealed that after 80 hours of ball milling the Rice Husk Ash particle size is reduced to nano range, which is an amorphous structure and is thermally stable. The nano Rice Husk Ash is successfully reinforced in molten aluminium 6061 using an electrical furnace equipped with stirrer and sonicator. Improvement in hardness of new aluminium 6061 nanocomposites is observed with the reinforcement of nano rice husk ash. It is concluded that the optimum percentage of nano Rice Husk Ash reinforcement in aluminium 6061 matrix is 2 percent in weight to obtain better hardness.

Keywords: DLS, DTA, EDX, Nano rice husk ash, Planetary ball mill, SEM, TG-hardness, XRD.

1. Introduction

Rice is the second largest standard diet for the people around the world. Paddy is widely cultivated in the East and South-East Asia to fulfil the need of the people in the world's highest populated countries such as India and China. Rice Husk (RH) is the outer membrane covering rice, which needs to be removed before it is being consumed. After milling the paddy, about 20% by weight of RH is removed and forms the by-product. On combustion, the dry RH gives about 14-20% of ash [1]. RH did not appear to have much value and is treated as agricultural waste. The burning of rice husk is an exothermic reaction and hence RH is widely used as fuel for small power plants and small industries such as parboiled rice mills, paper mills and sugar plants for steam generation and heating purpose. Rice husk possess good thermal insulation, good calorific value, flowability and is lighter in weight. Hence, it is very suitable for fluidized bed combustion boilers. After combustion of rice husk, grey coloured ash is produced and is called Rice Husk Ash (RHA), which is an industrial waste. If the rice husk is completely combusted, white coloured ash is produced. The husk contains about 75% organic volatile matter by weight, and the balance weight 25% is converted into ash during the firing process. This ash is known as RHA. Approximately, 20 g of RHA is obtained for every 100 g of rice husk.

Nowadays researches preferring natural fibers from agricultural waste such as corn stalk [2], coir fiber [3], RHA [4], etc., as a green initiative. The recent research studies reported that RHA contains around 92% of silicon dioxide (SiO_2), which is in amorphous form depending on burning temperature. It is further reported that the RHA can be used as reinforcing element in several applications such as metal matrix composites [5, 6], ceramic composites [7] polymer composites [8], and in green concrete as admixture [9]. Especially aluminium based metal matrix composites are gaining importance in replacing the monolithic counterparts in the field of automobile, aeronautical and space applications because of their low cost and isotropic properties [10]. If the reinforcing additives are in nano-size, then the resulting composites are called nanocomposites. It is already proven that the addition of relatively small amounts of reinforcing materials in nano-sizes gives good results than adding the same materials in micro size. Nanocomposites are gaining importance in overcoming the limitations of traditional materials and also composite materials [11]. Moreover, they can meet the challenges of the modern engineering applications.

Murthi et al. [12] made an attempt to modify the micro-sized fly ash into nanostructured fly ash using high-energy ball mill and characterized the samples. They proved that nano fly ash with AA2024 matrix improved the hardness and compressive strength.

Simoes et al. [13] investigated the influence of the dispersion technique for producing Carbon Nano Tubes (CNT) in the production of aluminium matrix nanocomposites. They found that addition of 0.75 % by wt. of CNT to aluminium matrix exhibited well dispersed and imparted highest hardness and tensile strength.

Ugheoke and Mamat [14] reviewed rice husk silica processing methods and properties. They concluded that the structure of the silica produced is not dependent on the purification of RH, but depends on the temperature of production.

RHA is a biomaterial, which is treated as agricultural and industrial waste. RHA is very rich in silica content. Several researchers are doing their research in

nanocomposites by selecting nano silica as reinforcing the material. In this paper, RHA in nano-size is chosen as reinforcing the material in the Al6061 matrix. As RHA is cheap and abundantly available in nature. Therefore, by utilizing RHA industrial or agricultural waste can become useful material and the cost of material production can be decreased.

In this paper, it is described that synthesis of white coloured NRHA and then characterization using Energy Dispersive X-Ray Analysis (EDX), X-Ray Diffraction (XRD), Dynamic Light Scattering (DLS), Scanning Electron Microscopy (SEM) and Thermogravimetric and Differential Thermal Analysis (TG-DTA). The synthesized NRHA is reinforced in aluminium 6061 (Al6061) matrix in various proportions and was tested for hardness. The results were shown the improvement in hardness of Al6061 when reinforced with NRHA.

2.Materials and Methods

Figure 1 summarizes the experimental procedure followed in this work.

To obtain standardized and repeatable results, it is beneficial to burn the rice husk under controlled combustion process. To undertake this study, rice husk was collected from a rice mill in Kakinada city in India. Kakinada city is located nearly at the centre of the east coast of India. The rice husk was thoroughly washed with tap water three times and then with distilled water once. The cleaned rice husk was then dried in sunlight. When observed with naked eye rice husk was in the shape of a ship hull and was in yellowish colour as shown in Fig. 2.

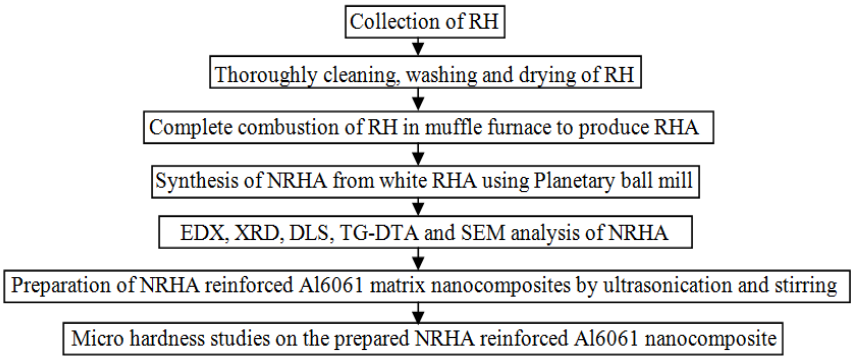


Fig. 1. Experimental procedure followed in this work.

Fig. 2. Cleaned, washed and dried RH, which is in yellowish colour.

2.1. Preparation of rice husk ash

Washed rice husk was then heated in an oven to 200 °C for 1 hr duration to remove the moisture and organic matter. During this operation, the colour of the husk was changed from yellow to black as in Fig. 3 due to the incomplete combustion of organic matter.

The ash disposed of by the industries, which are using Rice Husk as fuel is mostly grey in colour. The reason is that the raw rice husk is openly packed and there is the passage of air in the gaps, which causes easy firing. During combustion, the ash packs closely and prevents the air to reach the inside portion. Thermal treatment was followed for thorough combustion. In thermal treatment, the sample was kept in a muffle furnace and was maintained at a temperature of 650 °C for one and half hour. After this operation, the colour changed from black to white as in Fig. 4. At the completion of this stage, the Rice Husk Ash retained its shape just like a ship hull.

Saravanan and Kumar [15] analysed the chemical composition of the RHA composition and given as 94.04 % of SiO_2, 0.25 % of Al_2O_3, 0.14 % of Fe_2O_3, 0.62 % of CaO, 0.44 % of MgO, 0.02 % of Na_2O, 2.49 % of K_2O and 3.52 % of loss of ignition [16, 17].

Fig. 3. Grey coloured incompletely combusted rice husk.

Fig. 4. White ash after thermal treatment.

2.2. Synthesis of NRHA

The NRHA retained its shape after controlled combustion in the muffle furnace but it seems to be very fragile. The ash was manually ground with a kitchen pestle and was observed in a scanning electron microscope. In the SEM image, it was

observed that the average particle size was in the order of micrometres. The micro-sized particles were reduced to nano-size using planetary ball mill. In ball mill 10:1 ball to Rice Husk Ash powder ratio was collected in the drum and was rotated at a speed of 250 rpm. The ball milling was carried out for a total time of 80 hours. The samples were collected in airtight vials, which are shown in Fig. 5.

Fig. 5. Vials with collected samples of ball milled RHA.

2.3. Characterization of NRHA samples

The ball milled Rice Husk Ash samples were characterized using Energy Dispersive X-ray (EDX), X-ray Diffraction (XRD), Dynamic Light Scattering (DLS), Thermo Gravimetric and Differential Thermal Analysis (TG-DTA) and Field Emission Scanning Electron Microscopy (FE-SEM).

Elemental analysis of NRHA samples were conducted using EDX. EDX spectroscopy was carried out with the help of Field Emission Scanning Electron Microscope (FE-SEM) JEOL India Pvt. Ltd., JSM-7100F model. An adhesive carbon tape is a stick on the brass mount and a thin layer of NRHA particles were coated on it. A thin film of gold was sprayed over the NRHA layer in a gold deposition machine. By keeping the samples in the observation chamber of the FE-SEM machine and EDX spectrum was taken. The same procedure was repeated for three more NRHA samples and found the same spectrum for all the samples.

The crystal structure of NRHA was determined by X-ray diffraction machine (XRD, Bruker D8 Model, Germany). The NRHA Sample was mounted over a glass slide and tested on Bruker AXS D8 Advance X-ray powder diffractometer with nickel-filtered *Cu-kα* as radiation source with λ=1.54056 *A* at an accelerating voltage of 40 kV and at a scan rate of 2 ^{0}/min. The XRD pattern of the NRHA particles was recorded in diffraction with an angle range of 0°-90°.

The particle size distribution and the average size of the ball milled NRHA particles were measured using dynamic light scattering device Horiba SZ-100. RHA samples were collected for every 5 hours of ball milling and were tested for their particle size. Ethanol was used as the dispersion medium. The sample was sonicated using ultra-sonicator to separate agglomerated particles. During particle size measurement, the laser beam is focused through the particles and scatters at an angle. The scattered beam angle is inversely proportional to the particle size. The histogram was taken and observed the mean value of the particle size. The milling samples were collected repeatedly at 5 hours of milling interval until the mean particle size was reduced to less than 50 nanometers.

Thermal analysis of the fresh RHA and NRHA particles were studied by using TG/DTA EXSTAR 6300, India. The samples of 25 mg were taken in a Platinum crucible and the analysis was carried out at a heating rate of 10 °C/min from room temperature up to 1000 °C while sending Nitrogen gas at a constant rate of 100 ml/minute.

The surface morphology of the produced NRHA particles were examined by Scanning Electron Microscopy. The samples for FE-SEM analysis were prepared by drying a dispersion of NRHA particles on brass holder stick with carbon tape and gold coat. The particles were imaged by Field Emission Scanning Electron Microscope (FE-SEM) JEOL India Pvt. Ltd., a JSM-7100F model with Schottky field emission gun at an accelerating voltage of 15 kV, 33000 × magnification and on different scales ranging from 1 mm - 100 nm.

2.4. Preparation of NRHA particulate Al6061 matrix nanocomposite

Several researchers attempted the usage of RHA to develop new hybrid MMCs [18-22]. However, the use of NRHA is not reported in the literature so far. One possible reason, for not exploring the applicability of NRHA used in MMC, is the difficulty of dispersion of NRHA with aluminium at temperatures higher than 700 °C. The RHA nanoparticles are in the nanometer size and it is extremely difficult to disperse them uniformly in liquid metal due to agglomeration and clustering. To avoid such problems, ultrasonic power operated sonicator [4] as shown in Fig. 6 was used to disperse the nanoparticles uniformly throughout the molten Al6061. NRHA particle reinforced Al6061 matrix nanocomposites of Al6061 with 1 wt% of NRHA, Al6061 with 2 wt% of NRHA, Al6061 3 wt% of NRHA, Al6061 4 wt% of NRHA and also Al6061 alloy was cast by pouring the molten metal in the metal moulds.

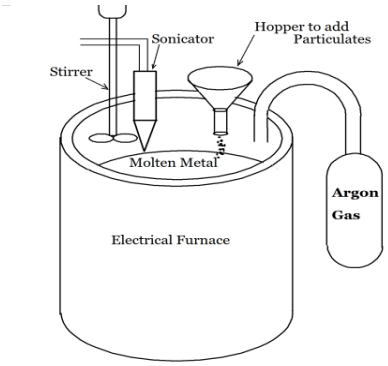


Fig. 6. Line diagram of electrical furnace with mechanical stirrer and ultrasonic power sonicator.

2.5. Hardness testing of NRHA reinforced Al6061 nanocomposites

The prepared NRHA particulate reinforced Al6061 matrix nanocomposites were cut to 50 mm square plate of 6 mm thick. The plates were ground by series of abrasive papers of increased grit size until the oxide layer is removed and then polished on a rotating disc polishing machine. These samples were tested for their hardness using DAKSH Vickers hardness tester, HVS-5 model.

3. Results and Discussion

The cleaned, washed and dried RH was completely burned in a muffle furnace at a temperature of 650 °C. The completely combusted RHA was milled in planetary ball milling with 10:1 ball to powder ratio at 250 rpm drum speed. Samples were taken periodically for every 5 hours and tested in particle size analyser. Milling is continued until getting nano-size particles. Nanoparticles of 43.8 nm mean size were obtained after 80 hours of ball milling. The prepared NRHA samples were characterized using EDX, XRD, particle size analyser, TG-DTA and SEM.

3.1. EDX analysis of NRHA

Energy dispersive X-ray analysis (EDX spectrum) of NRHA was carried out by FE-SEM with an EDX unit. Table 1 shows their relative percentages in the sample and Fig. 7 shows the EDX spectra of NRHA particles. The EDX of NRHA contained a very high percentage of oxygen and Silicon. Other elements such as Na, Mg, Al, K, Ca and Fe were in very small percentage. There was no presence of carbon peaks, which indicates complete combustion of RH. The EDX spectra indicate that the NRHA particles are stoichiometric. The weight percentages of silicon and oxygen were calculated from EDX data and found that Oxygen was 57.63 wt% and Silicon 37.8 wt%. The EDX graph shows that the chief constituent of Rice Husk Ash is silica.

Table 1. Chemical composition of NRHA.

Element	Weight %	Atomic %
O	57.63	71.51
Na	0.53	0.21
Mg	0.3	0.34
Al	0.35	0.05
Si	37.8	26.81
K	0.97	0.33
Ca	1.49	0.64
Fe	0.93	0.11

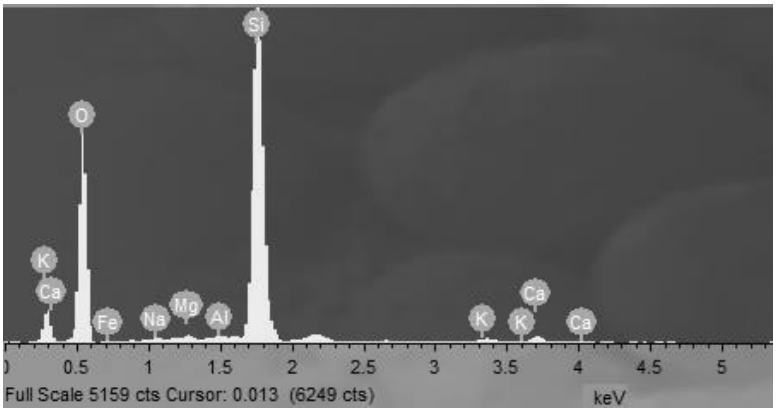


Fig. 7. EDX of NRHA particles.

3.2. Structural analysis by XRD

Figure 8 shows the X-ray diffraction graph for NRHA particles. The broad peak at 22° on 2θ axis shows that the structure of RHA produced is amorphous in nature [15, 16].

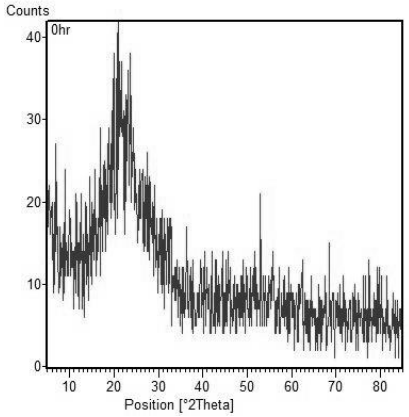


Fig. 8. XRD of rice husk ash.

3.3. Particle size analyser

The particle size of ball milled NRHA sample was done by particle size analyser (Nanoparticle size analyser Horiba SZ-100). Figure 9 represents the Dynamic Light Scattering (DLS) histogram of Rice Husk Ash particles. The size of the prepared nanoparticles can be observed in Fig. 9, which shows the mean particle size as 43.8 nm.

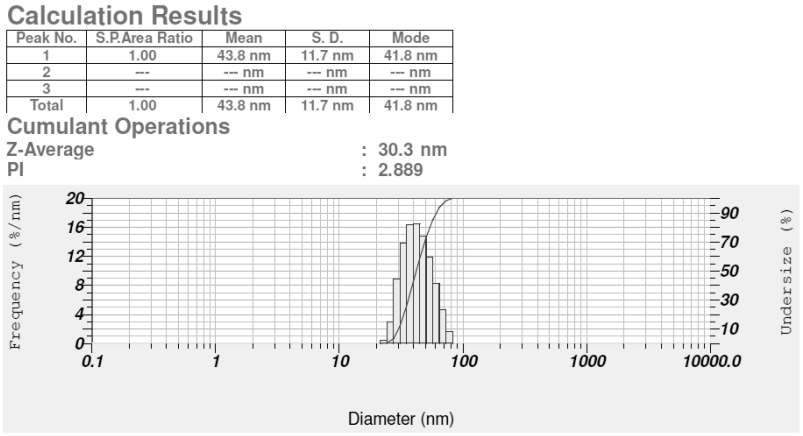


Fig. 9. Histogram of particles distribution of Rice Husk Ash nano particles.

3.4. Thermal properties of rice husk ash

TG-DTA is a thermal analysis technique, which is used to measure the loss of weight in a material with respect to increase in temperature and time. This experiment is very beneficial to investigate the thermal stability of a particular material. This experiment has to be conducted in a controlled environment from room temperature to 1000 °C. Prior to thermal analysis, the samples of RHA and NRHA were dried in a woven at

100 °C for a day. Approximately 25 mg samples of RHA and NRHA samples were placed in a Platinum crucible and kept in EXSTAR 6300 Thermogravimetric/Differential thermal analyser. The temperature was allowed to rise at a constant rate of 10 °C /min. While heating Nitrogen gas was continuously fed at a rate of 100 ml/minute. Heated Al_2O_3 was used as the reference sample. The loss of weight against an increase in temperature was recorded.

From Figs. 10 and 11, the loss in weight of the RHA and NRHA samples can be found with an increase in temperature and time. It can be observed that the weight loss of the material is around 4% between the temperatures 0 °C -150 °C. This weight loss is mainly due to vaporization of moisture content in the sample. From 150 °C to 650 °C, the loss of weight is around 3%. This loss of weight is due to the release of volatiles. From 650 °C to 1000 °C, there is no much weight loss and is recorded as 0.5%.

From Fig. 10, the total weight loss of the micro level RHA sample (un-milled RHA) was calculated as 10%. From Fig. 11, the total weight loss of NRHA sample is calculated as 7%. The TGA results showed that the weight loss of the nanomaterial sample is slightly less than the macro sample. Though there is no much difference between the two samples, it can be concluded that the material is considerably thermal stable at the nano level.

The Difference Thermogravimetric (DTG) curve of Fig. 11 shows it is exothermic up to 200 °C and after that, it is endothermic.

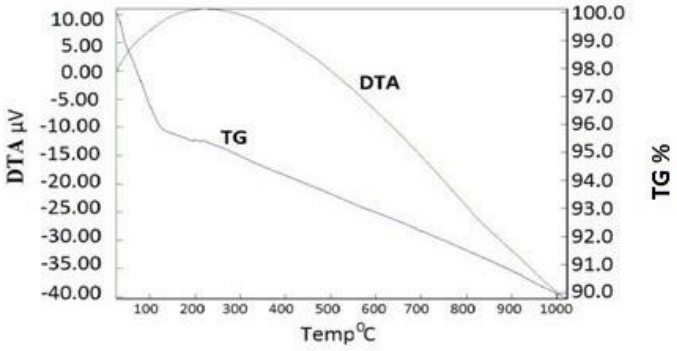


Fig. 10. TG-DTA graph for micro Rice Husk Ash sample (un-milled sample).

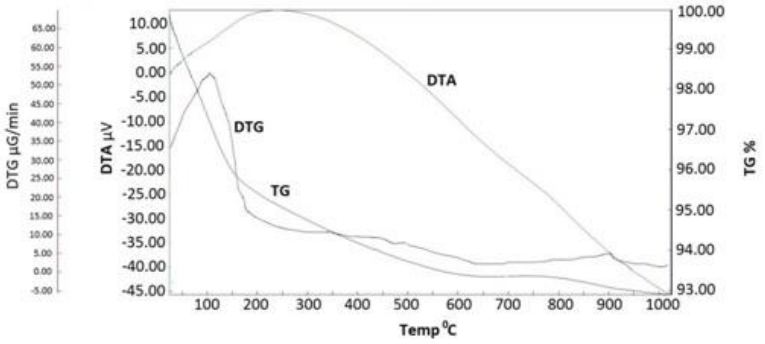
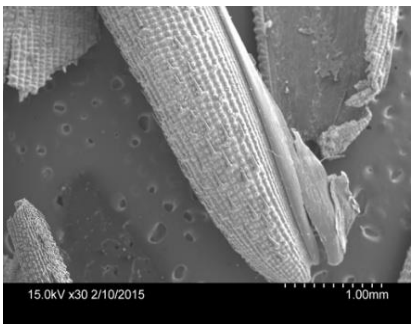


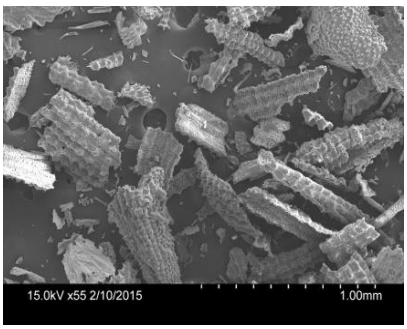
Fig. 11. TG-DTA graph for NRHA sample.

3.5. Morphology studies using SEM images

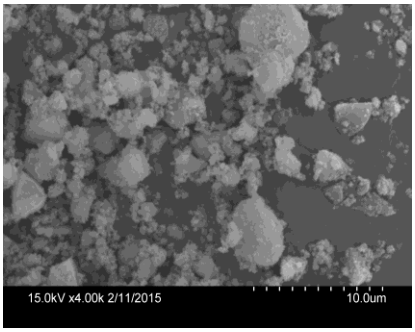
Figures 12(a) to (f) represent SEM images of RH and un-milled RHA and NRHA. The fresh Rice Husk Ash retained its shape but fragile in nature. SEM data revealed the high tendency of agglomerations of Rice Husk Ash nanoparticles. After 80 hrs of milling in planetary ball mill, irregular and very rough surface morphology was developed. There is the presence of some more micro size particles. This may be due to the agglomeration of nanoparticles or due to the limitation of ball milling, as the ball milling cannot produce uniform size particles.



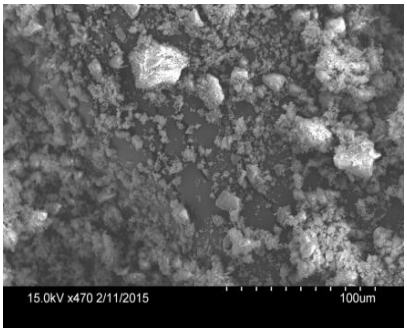
(a) Rice husk.



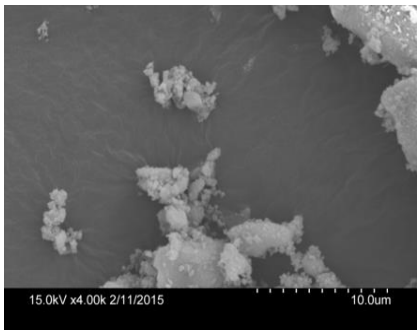
(b) RHA before ball milling.



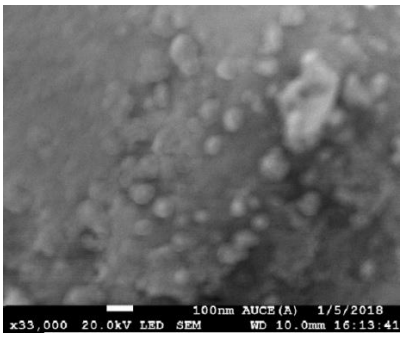
(c) RHA after 10 hrs of ball milling.



(d) RHA after 35 hrs of ball milling.



(e) RHA after 60 hrs of ball milling.



(f) NRHA after 80 hrs of ball milling.

Fig. 12. SEM images.

3.6. Micro hardness studies

Casting samples of NRHA particulate reinforced Al6061 matrix nanocomposites were prepared by using an electric furnace with stirrer and sonicator. The samples of size 50 mm square were taken for hardness testing. The ground and polished samples were tested using DAKSH Vickers hardness tester HVS-5 model, which is shown in Fig. 13. In order to eliminate possible errors, a minimum of five hardness readings was taken at different locations of each sample and their average values were tabulated. It is observed that the readings at different locations of each sample are more or less equal.

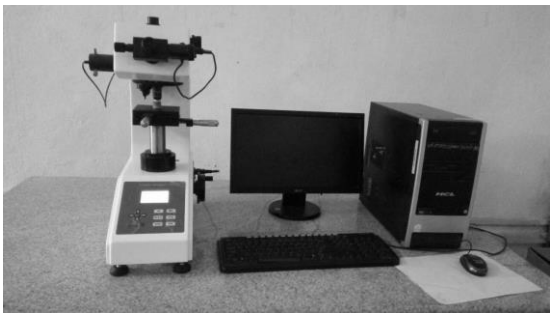


Fig. 13. Vickers hardness testing machine.

Hardness is the measure of resistance offered by the material for the local compressive load. It consists of a diamond indenter, in the form of a right pyramid shape with a square base and angle between the opposite faces is $136^{0.}$ A load of 500 gm was applied for 15 seconds. The indentation is shown in Fig. 14. For calculating Vickers Hardness Value (HV), the distance between diagonal corners has to be measured. The Vickers hardness number is the ratio of the load applied in kg to the area of the sloping surface of indentation in square mm. The formula is given in Eq. (1).

$$\text{Vickers Hardness } (HV) = \frac{2F \sin\frac{136^0}{2}}{d^2} \quad (1)$$

where d = Arithmetic mean of the two diagonals, d_1 and d_2 in mm.

The samples were tested and given as 79.5% of Al6061 alloy, 115.2% of Al6061 alloy with 1 wt% RHA, 124.6% of A16061 alloy with 2 wt% RHA, 114.3% of A16061 alloy with 3 wt% RHA and 82.7% of A16061 alloy with 4 wt% RHA were tested in Vickers microhardness testing machine.

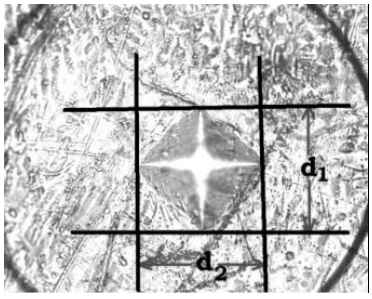


Fig. 14. Image of an indentation.

These results were plotted in Fig. 15. The Microhardness of aluminium alloy is increased from 79.5 to 115.2. Addition 2 wt% of NRHA in aluminium alloy resulted in a still higher value of 124.6. However, 3 wt% of NRHA reinforcement reversed the trend and the micro-hardness fell from 124.6 to 114.3. It may be noticed that this value (114.6) still represents a much higher Vickers micro-hardness value than the parent metal. Further addition decreased the hardness drastically.

A maximum of around 56 percent increase in hardness is recorded with the composite of Al6061 reinforced with 2 wt% NRHA. As the NRHA consists of rich Silica content, the NRHA definitely get the hardest structure. So the increase in hardness is primarily due to the refined grain structure of the matrix due to the presence of much harder NRHA. Furthermore, the nano-reinforcements strengthen the matrix by the creation of high-density dislocations during solidification of composites [12].

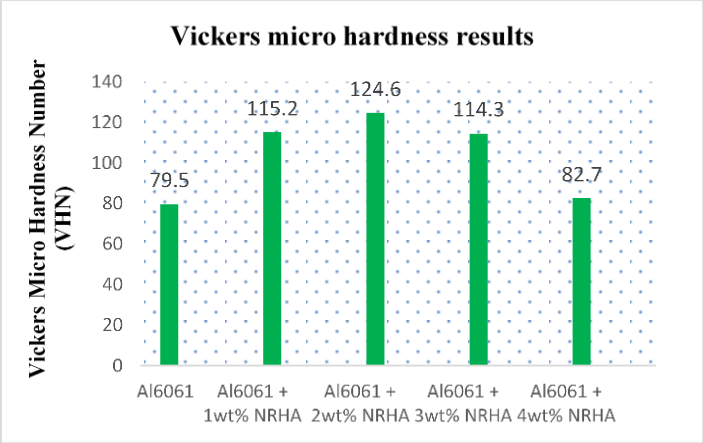


Fig. 15. Vickers micro hardness of aluminium alloy 6061, Al6061with 1 wt% NRHA, 2 wt% of NRHA and 3 wt% of NRHA.

4. Conclusions

The following conclusions were drawn from the present work:

- By the use of planetary ball mill, NRHA can get after 80 hrs of ball milling with 10:1 ball to powder ration and rotating at 250 rpm.
- From EDX analysis, the major constituent of the Rice Husk Ash is SiO_2.
- From XRD pattern, it is concluded that the crystal structure of the Silica in Rice Husk Ash is amorphous in nature.
- With TG-DTA it is concluded that the weight loss of RHA is approximately 10% and is not much varying with the reduction of the particle size. It shows NRHA is thermally stable.
- SEM images show that the surface morphology of ball milled NRHA particles is irregular and is very rough.
- Al6061 with reinforcement of 1-3 wt% NRHA improved the hardness of the prepared nanocomposite material, but above 3 wt% decrease in hardness of the casting is observed. Therefore the optimum percentage of NRHA reinforcement in Al6061 can be said to be 2 wt% to get good hardness values.

Acknowledgements

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Nomenclatures

d	Arithmetic mean of two diagonal dimensions, mm
d_1, d_2	Diagonal dimensions of indentation, mm
F	Applied load in Vickers hardness tester, kg

Greek Symbols

2θ	Position scale on horizontal axis of XRD image, deg

Abbreviations

CNT	Carbon Nano Tube
DLS	Dynamic Light Scattering
EDX	Energy Dispersive X-ray analysis
FE-SEM	Field Emission Scanning Electron Microscope
HV	Vickers Hardness Value
MMC	Metal Matrix Composite
NRHA	Nano Rice Husk Ash
RH	Rice Husk
RHA	Rice Husk Ash
SEM	Scanning Electron Microscopy
TG-DTA	Thermo Gravimetric and Differential Thermal Analysis

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