Study on Mechanical, Thermal and Morphological Properties of RHA Filled PVC Composite

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Abstract

Rice husk ash (RHA) and polyvinyl chloride (PVC) composite has been prepared by solution technique. Cyclohexane has been used as a solvent for PVC. The influence of RHA on different properties of PVC/RHA composite has been studied. The dispersion of RHA in PVC matrix has been evaluated by the SEM studies. The thermal properties showed an increasing trend as the filler concentration is increased in the PVC matrix system. The melting and crystallization behavior has characterized by DSC analysis. Thereafter, the mechanical properties like tensile strength showed an increasing nature when composite comprise the 90:10 of RHA and PVC in the composite. Modulus has shown best results at the 70:30 due to hard and strong nature of composite. Elongation at break found maximum of virgin PVC. The hardness of the prepared composite has been increased when loading has been increased of RHA in PVC. Furthermore, this research work has shown that the solution technique is easy and cheap method to produce PVC/RHA composite for different applications.

Keywords: Mechanical properties, Rice Husk Ash (RHA), Polyvinyl Chloride (PVC), Thermal & Morphology, Fourier Transform InfraRed Spectroscopy (FTIR), Composite

Introduction

Rice hull is an organic waste which is used as fuel to generate steam. Rice hulls contain about 75 % organic volatile matter and the remaining 25 % of the weight is converted into ash. RHA comprises high amount of silica 92-95 % of total weight. Additionally, high porosity and light weight with very high surface area is essential requirement for filler in composites. Environmental safety from RHA is a great challenge for researchers and scientists.[1] In a study S.P. Deshmukh and A.C. Rao prepared a composite of PVC and mica. The results shows a better in dielectric properties with improvement in young's modulus shore D hardness and stiffness, the elongation at break is decreased. Due to the lower in particle size

RHA decreases the volume of large pores and therefore makes the continuous pores into discontinues ones. This will leads to make the structure more homogeneous and denser of the matrix system.[2, 3] The large number of rice husk ash has been used as a pozzolana in various countries due to lower in cost and high reactivity with plastics and cements. [4] When burning at 600 to 700^{O}C temperature for 2 hours RHA contains 90-95% SiO_2 content, amorphous nature and develops cellular form, with 50-1000m2/g surface area. [5]

Azman Hassan et al [6] concluded that the addition of RHA in PVC-U increased the fusion time. It is also revealed that the RHA and PVC-U composite with shorter fusion time and fusion torque was increased to the higher point. It is well evidenced in this paper that upon the addition of 20 phr RHA, the end torque is decreased compared to unfilled sample. Polyvinyl chloride (PVC) is a product based on chlorine and ethylene that can be combined to form the monomer vinyl chloride. PVC is a thermoplastic material that can undergo softening and cooling many times without any significant chemical change. PVC has a good combination of stiffness and impact strength with the aid of non-flammability. Including these properties and applications in the construction, electrical, automobile and medical industries it is chosen as the base matrix material as a binder for the RHA filler.

Rice husk is a low valuable agricultural by-product produced by the rice mills because of exceeding in utilization and production of rice in the developing countries. Due to the high silica content it is used as filler in the polymers, rubbers industries and cement products for reducing the cost and increasing the strength. [10]

There are so many papers can be found on rice husk with the other polymeric materials [1,7,13]. Maleic anhydride modified rice husk filled PVC composite, Carbon /silica composite, blended with cement for increasing the strength and due to the low thermal conductivity, high melting point, low bulk density and high porosity makes it fine filler for the industries. [6-17]

In this research, we are trying to develop a new kind of composite based on polymer and RHA for its various applications in the field of desiccant polymers, electrically insulating and other places.

2. Materials and Experimental Methods

2.1 Raw Materials

The following materials listed in table 1 were used in the preparation of the composites. Polyvinyl chloride (PVC) was obtained from Research & Development, CIPET Lucknow. Rice Hush was obtained from the local rice miller situated in Lucknow, Uttar Pradesh. Cyclohexane was supplied from Lova Chemicals Private limited, Mumbai.

Table 1 Materials and suppliers with specification.

Materials	Specifications	Suppliers
Poly Vinyl Chloride (Resin)	Density:1350-1700kg/m^3	Research & Development Lab., CIPET, Lucknow
Rice Husk	Bulk Density:70-80g/cm^3 Length: 3-6 mm	Local rice miller in Lucknow, Uttar Pradesh.
Cyclohexanone	Density: 0.9478 g/mL (liquid)	Lova Chemie Private limited, Mumbai.

2.2. Experimental Methods

2.2.1. Preparation of Rice Husk Ash (RHA)

The collected rice husk from a local rice mill contains many impurities like dust, small rice particles, and fine sand particles. These impurities could be influenced the properties and composition of the rice husk ash, so it need to be cleaned to get pure rice husk. This purpose could be attained by washing the rice husk thoroughly with the double distilled water. Some extra care was taken so as the rice husk was not washed out in this process. This kind of wash removes major impurities like sand particles or dust particles & small rice particles remained in rice husk. The finally obtained rice husk kept all day in the sun to dry it up. The husk was thoroughly dried so as no amount of moisture remains in this process and desiccator was used to do not introduced moisture till the preparation of composite. Finally, the rice husk ash was produced by burning rice husk at 650^{O}C in a furnace for 30 minutes burning time. After the whole process the obtained rice husk was sieved to get the uniform particle size and lesser particles (in micrometer) of rice husk ash as filler.

2.2.2. Composite Preparation

Poly vinyl chloride, Rice Husk Ash (RHA) and Cyclohexanone were used in the preparation of PVC/RHA composites. Furthermore, Rice Husk Ash was sieved in a sieve analyzer with the different mesh size upto 240 mm were used after the preparation steps of rice husk ash. The solution blending technique was used to prepare the composite of PVC/RHA with the different loadings of rice husk ash (10, 20, and 30) in weight %. Pre-dried rice husk ash was kept in a desiccator to remove vacant moisture. The mixture of PVC and RHA were taken simultaneously in a beaker capacity of 1 liter and cyclohexanone was used as a solvent to dissolve them with the help of a glass rod and stirred thoroughly for half an hour to get dissolve PVC and allow to introduce RHA particle in the PVC matrix. The preparation of composites has been completed with the help of compression molding machine. Before the mixtures were poured inside the mould, the mould was initially polished with silicon as a release agent to prevent the composites from sticking to the mould upon removal. After the mixture had been poured into the mould, it was consolidated by compression moulding at room temperature for 24 hours for fully cured and hardened at the load of 2.4 MPa.

2.2.3. Testing & Characterization methods

Mechanical properties of the samples, such as tensile strength, tensile modulus, elongation at break, and flexural strength were measured according to the ASTM D-668 standards with the aid of universal testing machine (UTM, Instrom) at room temperature (23^{O}C) and crosshead speed of 5 mm/min., all the samples were cut down as the ASTM standards. Furthermore, thermal studies were carried out by Differential Scanning Calorimetry (DSC; Perkin Elmer) and Thermogravimetric analysis (TGA) in the temperature range from room temperature to 300^{O}C at a constant heating rate of 10^{O}C/min and 50 to 700^{O}C, respectively. Fourier transform infrared (FTIR), ATR spectroscopic studies were carried out on the composite samples with a Perkin Elmer FTIR instrument. The graph was plotted by PerkinElmer Spectrum (version 10.03.06). Thereafter, Scanning Electron Microscopy (SEM) was used to study the surface morphology of the developed composite. Micrographs were taken out at 2.00 KX resolutions at the voltage of 15kV. Before taking the micrographs all samples were coated with gold in an auto fine coater (JOEL, JFC-1600) to avoid the charging effect and increase the efficiency of secondary electrons.

3. Results and Discussion

3.1. Mechanical properties

3.1.1. Tensile Strength& Tensile Modulus

In table 2 and figure 1 & 2 tensile strength and tensile modulus results of the developed PVC/RHA composite have been shown. It can be observed from the test results that the addition of rice husk ash filler decreased the tensile strength of the neat PVC. However, there is a different trend of decrement in the tensile strength as, when the 10% of RHA is loaded into the PVC matrix the tensile strength reduced from 45.2 to 28.1 MPa and thereafter a gradual decrement from 25.8 to 20.7 when loadings of RHA are 20 and 30 wt%, respectively. From these results the effect of RHA at different loading into PVC matrix has been clearly shown. The sharp decrement in the tensile strength of PVC/RHA composite can be attributed to that the morphology of the blends revels the poor and weak adhesion between the polymer matrix and rice husk ash filler and large cavities or cracks also influenced the tensile strength of the developed composite of RHA/PVC.

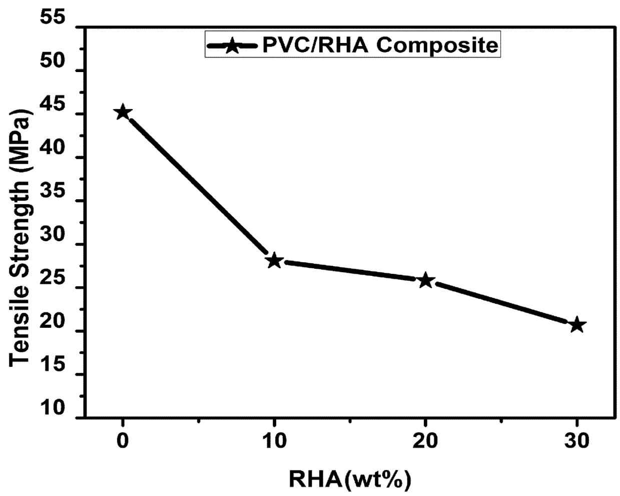


Figure 1 shows tensile strength of PVC/RHA composite.

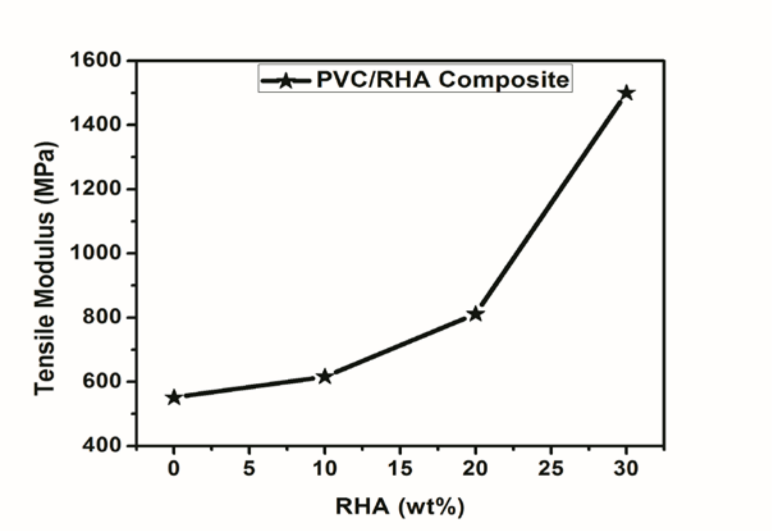


Figure 2 shows tensile modulus of PVC/RHA composite.

Generally the rigidity of the organic materials has lower side when compared to the inorganic fillers. Therefore, the addition of these rigid particles into the PVC matrix results the significant improvement in the tensile modulus and decrease in the tensile strength. The rigid and stiff nature of the composite correspondingly decreases the tensile strength and increases the tensile modulus.

Table 2 shows the mechanical properties of PVC influenced by the RHA

Sample	Composite designation	Tensile strength (MPa)	Tensile Modulus (MPa)	Elongation at break (%)	Flexural strength (MPa)
1.	P_{100}	45.2	551	80	95
2.	$P_{90}R_{10}$	28.1	616	69	60.9
3.	$P_{80}R_{20}$	25.8	811	30.7	48
4.	$P_{70}R_{30}$	20.7	1500	28	40.6

3.1.2. Elongation at break

It has been seen in the table 2 and figure 4 that there is a decrement in the elongation at break of the test samples, increasing the loading of RHA filler in the PVC matrix. Due to the addition of rice husk ash the movement and displacement of the polymer chains are restricted. SEM micrographs revealed that the bonding or adhesion between the RHA filler and PVC polymer matrix is weak and those cracks and multiple surface structures reduced the elongation at break of the PVC/RHA composite. [2]

The neat PVC retained 80% elongation at break, while 10 wt% of RHA reduced it to 69% in comparison to neat PVC. Furthermore, a sharp change (from 69-28%) in the elongation at break has been observed when the filler content is increased upto 30 wt% of RHA in the polymer matrix. The rigid nature of the RHA filler is due to the higher silica content in the ash and the rigidity of the RHA filler also improved the stiffness and rigidity of the composite system.

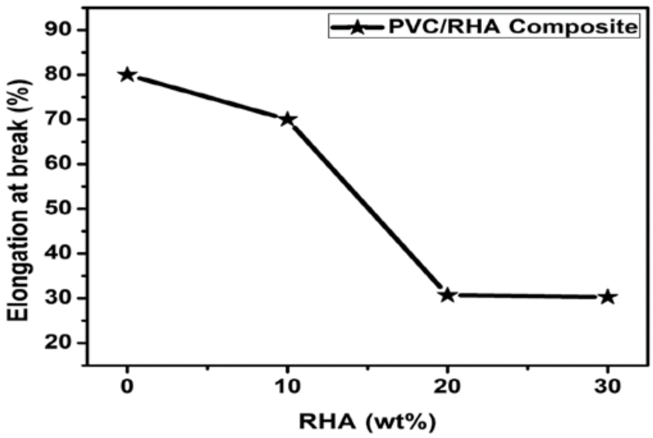


Figure 3 shows elongation at break of PVC/RHA composite.

3.1.3 Flexural Strength

The flexural strength of the PVC/RHA composites is presented in the table 2. Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. Therefore, it is depends on the bonding and adhesion between the filler and the matrix of the composite system. The dispersion of the filler also plays a key role to give the strength to the composite. In figure 4 it is clearly shown that the addition of the 10 wt% of filler sharply decreased the flexural strength of the PVC/RHA composite. Furthermore, it is revealed that the filler content upto 30 wt% not significantly decreases the flexural strength.

However, in our study the flexural strength of the PVC/RHA composite decreased while increasing the content of the RHA. This may be attributed due to the agglomeration of the RHA particles in the developed composite.

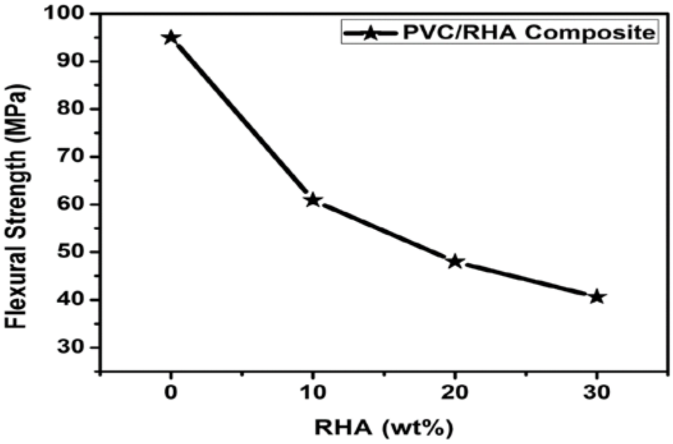
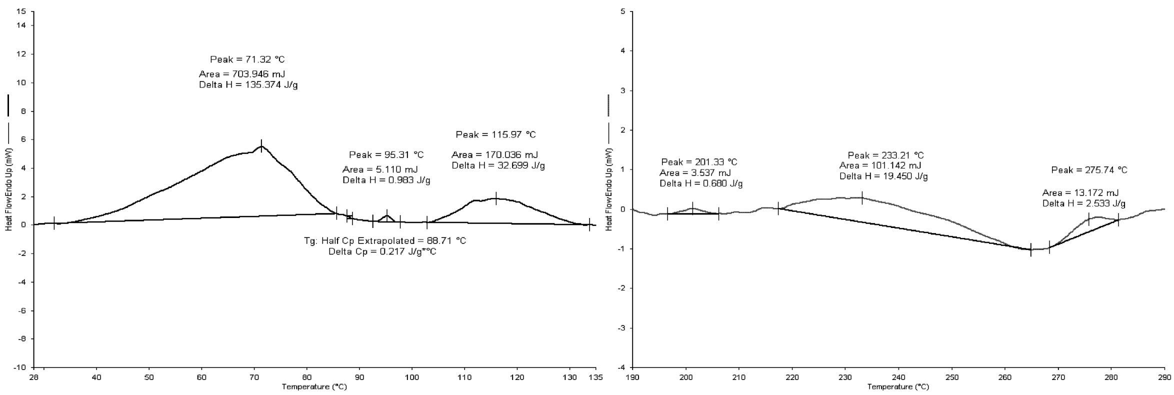


Figure 4 shows flexural strength of PVC/RHA composite.

3.2. Thermal properties

3.2.1. Differential Scanning Calorimetry (DSC)

The DSC thermograms illustrated in table 3 and figure 5a-d. The exceptional thermal properties of prepared PVC and RHA based composite has been harness as fillers to improve thermal stability and dimensional stability. The melting point (Tm) of virgin PVC material is shown in figure 5a. The results revealed that virgin PVC shows three peaks at 71, 95, 115^{O}C. The Tm of PVC/ RHA composite was higher than that of virgin PVC. The melting point peaks for PVC and 10 wt% RHA composite found to 201, 233, 275^{O}C due to the small incorporation of RHA in the PVC matrix. Furthermore, as be amount of filler was in increased to 20wt% the melting point of PVC/RHA composite was increased 295 and 320^{O}C.



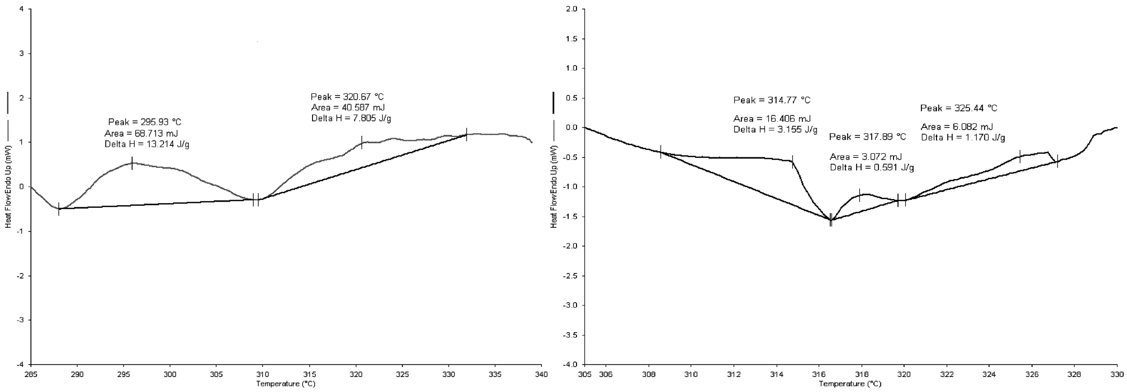


Figure 5 (a-d) shows the DSC thermo grams for different PVC/RHA composite system.

But when the amount of filler was increased upto 30 wt% in the PVC matrix, the melting point for first second and third peaks were higher than the other composite compositions and found 314, 317 and 325^{O}C. Therefore the use of RHA particles as filler affects the melting behavior of the develop composites. Bahareh Azinfar et al concluded that, the probable hindrance of polymer chains movement due to the RHA filler particles can restricted the change from crystallization. The higher melting temperature of PVC/RHA composite compared to virgin PVC can be summarized by the slow heat transfer in the composite through RHA particles.

Table 3 refers the thermal data of different formulations.

Sample No.	Melting point (^{O}C)			ΔH (J/g)		
	Peak 1	Peak 2	Peak 3	Peak 1	Peak 2	Peak 3
1.	71	95	115	135.3	0.98	32.6
2.	201	233	275	0.68	19.45	2.53
3.	295	320	-	13.21	7.80	-
4.	314	317	325	3.15	0.59	1.17

3.2.2. Thermogravimetric analysis (TGA)

The influence of rice husk ash (RHA) on the thermal degradation or stability of PVC was assessed by TGA in table 4 and figure 6. The TGA curve suggested that the addition of rice husk ash as a filler in the PVC matrix greatly increased the thermal stability and weight loss was also increased due to the higher temperature of rice husk ash. The final degradation temperature (at 40 wt% loss) of the different composite systems recorded as given in table. We resulted that the at the 10 wt% of loading of RHA the higher thermal stability is achieved (684^{O}C) due to the even and fine dispersion of filler with the polymer matrix. The initial degradation temperature (IDT) at 10 wt% loss was decreased upto 63 ^{O}C and the degradation at 40 wt% loss was achieved 355^{O}C. S. Kumar et al showed this kind of trend in carbon/polyetherimide composite which reduces the thermal stability of the composite system. [19] The addition of RHA upto 30 wt% also increased the final degradation temperature (FDT) of the developed composite which revealed the degradation at 30 wt% was maximum (686 ^{O}C) for the composite. By the SEM micrographs it is concluded that the addition of 30 wt% RHA in the PVC matrix has fine dispersion of filler and the improved adhesion between these components. Lower amount of cavities and distorted surface also improved the thermal stability of composite system. Navin Chand et al reported that the three types were achieved during the thermal degradation process. As compare to this, the same trend was shown in the present study and degradation of PVC was influenced by the RHA as shown in above results. The degradation at the range between 500-700 ^{O}C was recorded due to the other compounds present in the RHA.

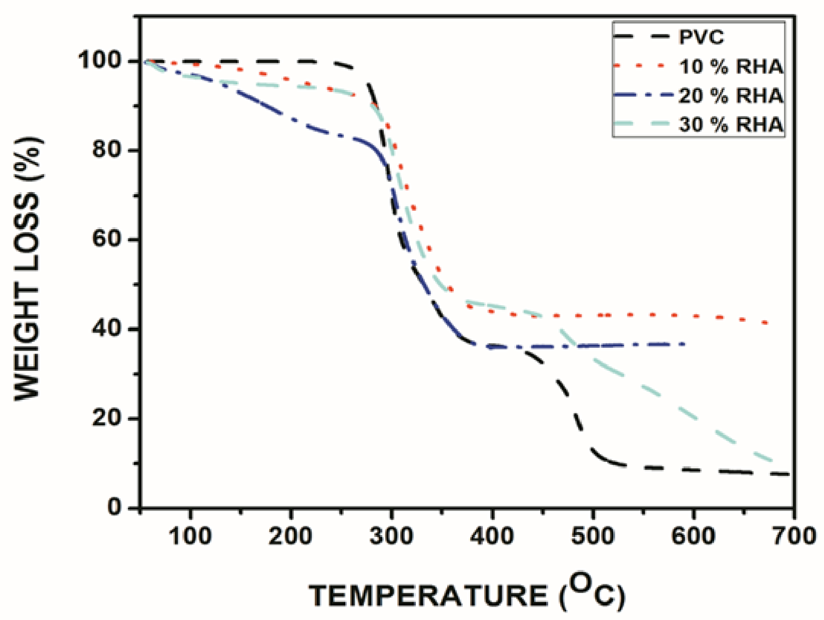


Figure 6 shows thermal stability of the PVC/RHA composite at different filler level.

Table 4 shows the degradation temperature of developed composites.

Sample designation	Initial Degradation Temperature (IDT)	Half Degradation Temperature (HDT)	Final Degradation Temperature (FDT)
P_{100}	253^{O}C	331^{O}C	358 & 521^{O}C
$P_{90}R_{10}$	116^{O}C	35 ^{O}C	684^{O}C
$P_{80}R_{20}$	64^{O}C	331^{O}C	398^{O}C
$P_{70}R_{30}$	63^{O}C	348^{O}C	686^{O}C

3.3. FTIR spectroscopy

Table 5 and Figure 6 shows the spectra of the developed PVC/RHA composites. In the FTIR results eight major peaks are observed as given in the table 3, respectively. In the FTIR transmittance plot of PVC/RHA composite a broad peak is observed at 3419cm-1, which corresponds to O-H hydroxyl bond and can be attributed to water observed by the composite system with transmittance of 36.6%. Another two sharp but narrow peaks are observed at higher wavenumber 3021 cm-1 and 2963 cm-1 with transmittance 15.2 and 29.3 respectively and corresponds to asymmetric and symmetric stretch bonds of C-H. The peak around 1386 cm-1 is assigned to C-H aliphatic bonding bond. The bonding bond of C-H near Cl generates the peak at 1215 cm-1 with transmittance level of 40.5%. The C-C stretch bond to PVC/RHA composite backbone chain occurs at 1074 cm-1 with transmittance of 42.7%. Due to the presence of rich silica filler Si-O vibration is recorded at 762 cm-1 with 29.5% transmittance. Finally, the peak at 670 cm-1 relates to C-Cl gauche bond with the 47.4 transmittance.

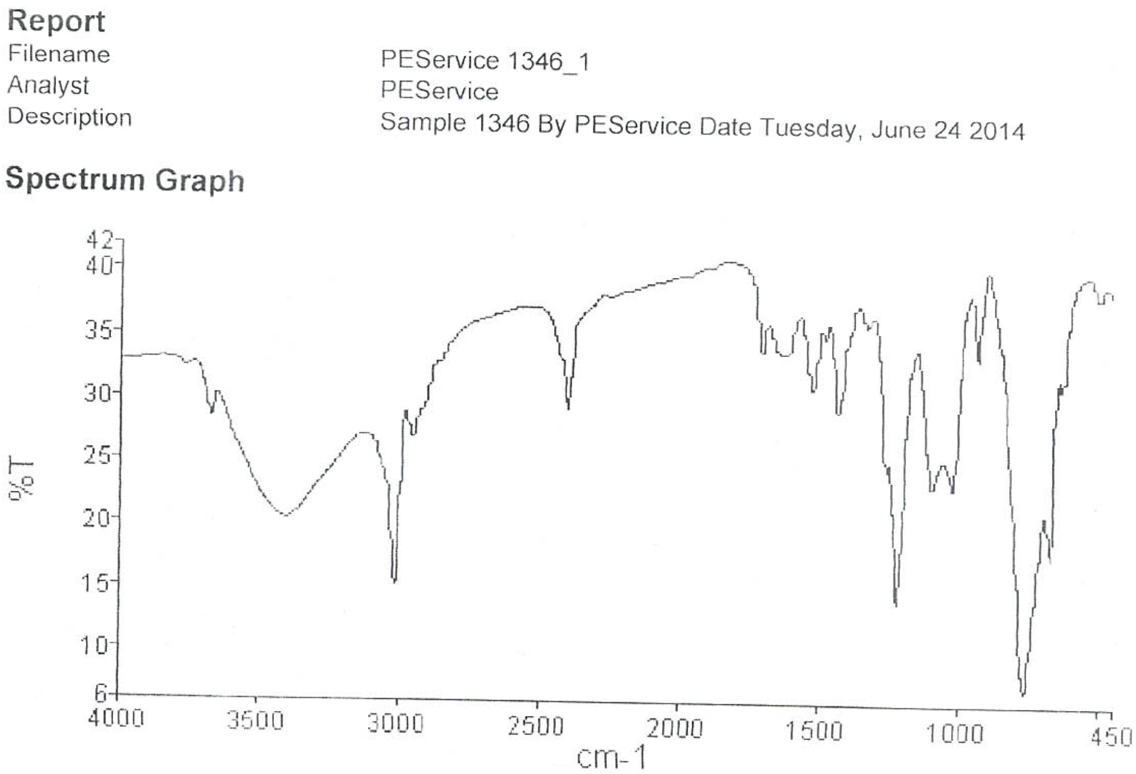


Figure 6 showing the graph plotted between wavelength (cm^{-1}) and transmittance (%) of the PVC/RHA composite.

Table 5 displays the peak values observed by the FTIR results.

S.No.	Wavenumber on X-axis (cm^{-1})	Transmittance Y-axis (%)
1.	670	47.4
2.	762	29.5
3.	1074	42.7
4.	1215	40.5
5.	1386	43.3
6.	1636	41.6
7.	3021	26.8

3.4. Morphological properties

Figure 7 presents the SEM images of the PVC/RHA composites with the different loadings of RHA. Rice Husk Ash (RHA) particles were embedded in the PVC matrix to yield composite with the loadings of 10 wt%, 20 wt%, and 30 wt% as presented in figure 7 (a-c). In the composites dispersion of filler can affect the properties. The figure 7 (a) indicates that the surface of the sample is relatively smooth and fine but large cavities, cracks and island like structures are present. The size of the cavities are large and sharp, showed a strong incompatibility and poor interfacial adhesion between PVC and RHA. The dispersion of filler and surface texture of RHA particles between the three composites have been seen. Figure 7 (b) and 7 (c) shows that the cavities are smaller when compared to the 10 wt% RHA filled PVC composite. The surface morphology of 20 and 30 wt% RHA filled composite present's uneven surfaces and pieces of filler which indicates poor adhesion and weak interaction between the filler and matrix. The significant decrement in the tensile strength and elongation at break occurs due to weak interaction of RHA to PVC and high level of particles pullout when samples were prepared and mechanical tests processed. [2]

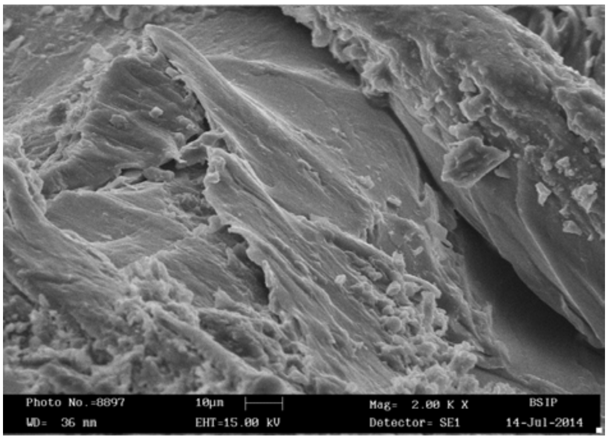


Figure 7 (a) shows the SEM micrographs of developed composite of PVC/RHA

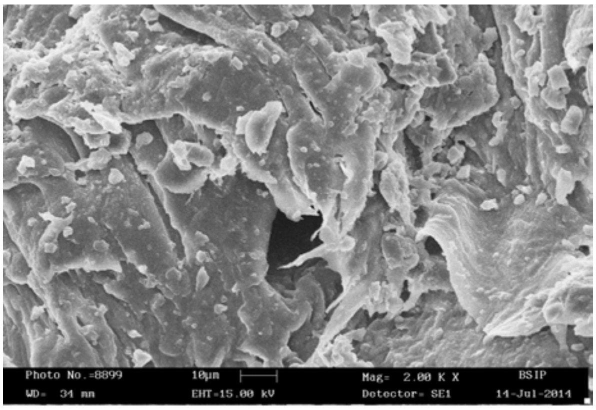


Figure 7 (b) shows the SEM micrographs of developed composite of PVC/RHA

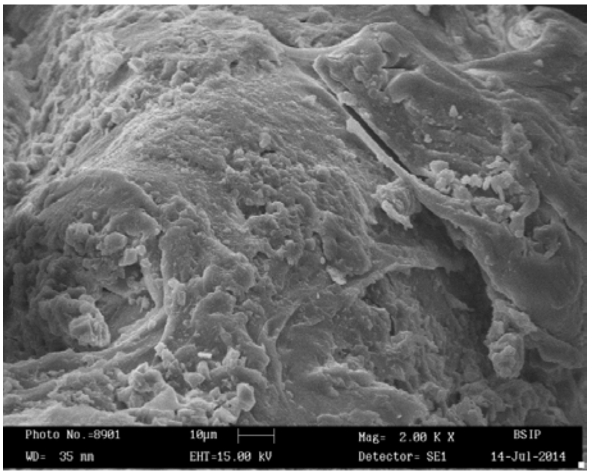


Figure 7 (c) shows the SEM micrographs of developed composite of PVC/RHA

4. Conclusion

Rice husk ash is used as reinforcing filler in the PVC matrix. The composite has been fabricated via solution blending method and concentrations of rice husk ash to fabricate the composites used as 10, 20, 30 wt % of the matrix. On the basic of above results it is conclusion is following that:-

1. Tensile strength, elongation at break and flexural strength has been decreased due the poor filler dispersion and interaction with the matrix. While the tensile modulus is increased due to the rigidity and toughness of the RHA filler provided to the matrix PVC.

2. Thermal property has been increased when the concentration of the filler is increased due to the higher temperature of rice husk ash RHA, the presence of the silica (SiO_2) and other elements and their higher amount of melting and ignition temperature increased the thermal properties.
3. FTIR and SEM studies have been accompanied to analysis of the functional groups present in the composite system and surface analysis. It is evaluated that the poor and weak filler and polymer matrix interaction and bonding leads to decrease the properties of the developed system.
4. The composite can be used where high temperature resistance and chemical resistance is required due to the PVC and RHA. Lower in cost and easy in the processing makes this way better for fabrication and developing and improves the properties of the composite by adding the coupling agents and grafted polymer.

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