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Natural Nanosilica from Agricultural Waste Rice Husk and Its Magnetic Composites for Its Possible Applications in Semiconductor, Catalytic Activity and Biomedical Science

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Abstract

This research demonstrates the sustainable synthesis of crystalline nanosilica (SiO_2) from rice husk, an agricultural waste, via a cost-effective route. The nanosilica was integrated with ferrites and polyvinylidene fluoride (PVDF) to create a magnetic nanocomposite using injection moulding equipment. X-ray diffraction (XRD) confirmed the crystallinity of the SiO_2, while scanning electron microscopy (SEM) revealed nanoscale particle morphology. Fourier-transform infrared spectroscopy (FTIR) identified Si–O–Si functional groups, validating the silica structure. UV–Vis spectroscopy showed strong UV absorption, indicating potential catalytic applications, and low visible light absorption. Tauc plot analysis yielded the direct and indirect band gaps. The indirect band gap was determined to be 1.73 eV, and subsequently direct band gap is 1.88 eV, which is similar to semiconductor electronics materials. Magnetic measurements showed diamagnetic behavior for pure SiO_2 and ferromagnetic properties for the SiO_2–ferrite–PVDF nanocomposite, exhibiting distinct hysteresis loops. These results highlight the successful valorization of rice husk-derived nanosilica from agriculture waste in producing polymer magnets with potential applications in semiconductor electronics, catalytic activity and biomedical science.

Keywords: Nanosilica, Rice Husk, Magnetic Nanocomposite, Magnetic polymer

1.Introduction

Metal oxides play an essential role in many areas of chemistry, physics, materials science, and related emerging technologies. Oxides are used in the fabrication of microelectronic circuits, sensors, piezoelectric devices, gas sensor cells, coatings for the passivation of surfaces towards corrosion, and as catalysts [1]. Oxide nanoparticles can show off special bodily and chemical properties due to their confined size and an excessive density of corner or surface sites. Hence, transition metallic oxides (TMO) are the most widely used in the emerging area of magneto-electronics, catalysts, and photo catalysis, solar cells, and gas sensors [2]. SiO_2 has been a valuable material due to its superb physical and chemical characteristics. It exists in many crystalline forms, the better known being quartz, cristobalite, tridymite, stishovite, and coesite; however, its best acknowledged shape is amorphous silicon dioxide. Silica nanoparticles has been explored for different aspects in accordance to its purity, shape, measurement, and distribution, and widely applied in the mechanical industry and as precise structures for chemical catalysts, ceramics, and photo-electricity elements, etc. Applied fields for the silica corpuscular powder are expanding regularly because of its physicochemical merits. Crystalline SiO_2 has extra sharp points in the interband transition electricity spectrum than amorphous SiO_2, the power of the absorption part for crystalline SiO_2 is about 1eV greater than that for amorphous SiO_2 [3,4]. The silicon dioxide has been synthesized via a number of techniques. There is considerable activity in the synthesis of crystalline and uniform material for the functions in microelectronics, optics, electrical, and several other fields. It has a pattern having high purity to discover achievable functions in many fields, such as managed launch applications, sensor units and catalysis, and dielectric substances [5]. The sensitized mesoporous silica with acriflavin dye should find purposes in Nano sensors and in Nano lasers [6]. The photocatalytic recreation of oxide materials and polymers can be elevated with the aid of the addition of SiO_2, which will increase the accessible surface area of the catalyst due to its giant band gap [7, 8]. The electromagnetic behavior of the ferrite magnetic material can be considerably tuned using different fillers such as SiO_2, TiO_2, MoO_3, etc [9] and due to the reason being these fillers promote the catalytic process for tuning the electromagnetic behavior. In recent years, studies have been carried out for the enhancement of the electromagnetic properties of ferrite magnetic material via using SiO_2 as filler, for example, $Ni_{0.65}Zn_{0.35}Cu_{0.1}Fe_{1.9}O_4$-$SiO_2$ [10, 11] $Co_{0.5}Zn_{0.5}Fe_2O_4$-SiO_2 [12], $Ni_{0.5}Zn_{0.5}Fe_2O_4$-SiO_2 [13-20], $Co_{0.84}Fe_{2.16}O_4$-SiO_2 [21]. The increasing demand for these materials in the areas of chemical, ceramic, chromatographic, coating, catalysis, energy, emulsifier, organic science, etc., needs the most efficient and dependable synthesis method for the production of these nano-composite materials. As per the reports, several synthesis techniques employed for the coaching of nano-composite materials are micro-injection moulding, in-situ polymerization, co-precipitation, sol-gel, and many others [22, 23]. Nanosilica and its composite materials have found widespread applications in various fields such as dental science, agriculture (as nanofertilizers), and targeted drug delivery systems due to their high surface area, biocompatibility, and ability to encapsulate active compounds and presence of Si-O chemical bond [24, 25, 26]. The crystalline nano silica powder is prepared by using the leaching process, which is a chemical-based technique. After preparation of this Crystalline Nano silica powder, it is used as a filler material for the production of a nanocomposite of nano silica. Thus, the present work aims is synthesize the crystalline Nano silica and the composite of PVDF/Nano silica/$Ni_{0.8}K_{0.2}Fe_2O_4$ successfully and used various characterization techniques to study its morphology, structure, magnetic and optical properties for its possible applications from semiconductor properties to magnetic polymer. Another objective of this research is to prepare functional materials at the nanometric scale from natural sources, from waste rice husk, and measure their physical properties for their applications and progress of research and development activities from electronics to biomedical science.

2. Materials and Methods

2.1 Preparation of crystalline Nano silica: Leaching and co-precipitation were used to make crystalline nano-silica from rice husk ash (RHA). Rice husk was obtained from a rice mill located Purnea distrct of Bihar, India and thoroughly washed with deionized water to remove the polluted materials (Merck, India). Rice that has been rinsed of its husk was dried in the sun for 12 hours before being carefully pulverized with a grinder machine for grinding. The crushed rice husk was first burned at 600°C for 2 hours before being cooled. In a Muffle furnace, at 1000°C for 2 hours. RHA was obtained and treated with 6 NH_4Cl (Merc, India) for 1 hour, 2 hours, 3 hours, 4 hours, and 24 hours, and during the leaching process, the mixture was agitated for 2 hours in a magnetic stirrer. The leached RHA was then filtered using Whatman Grade 1 filter paper and deionized water. The filtration procedure was continued until the pH of RHA reached 7. The neutralized RHA was incubated in 2.5 N NaOH solutions for 2 hours at 80°C with a magnetic stirrer. The aforementioned solution was then neutralized again with warm deionized water, yielding sodium silicate. The details of the relevant reaction are as follows:

$$\mathbf{SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O}$$
$$\mathbf{Na_2SiO_3 + 2HCl \rightarrow SiO_2 + 2NaCl + H_2O}$$

The sodium silicate was then treated with concentrated HCl until the pH reached 2, resulting in the precipitation of amorphous Nano silica. The extracted Nano silica was then annealed in a muffle furnace for 1 hour at 1000 degrees Celsius. In the end, crystalline Nano silica was produced. The schematic diagram for preparation is shown in Figure 1.

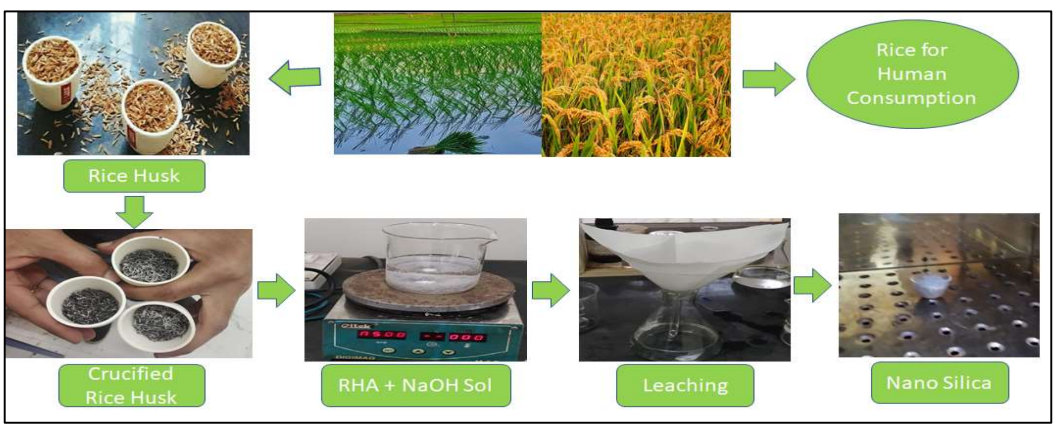


Figure 1: Flow chart of synthesis process of production of Nano silica from rice husk.

2.2 Preparation of Magnetic composite materials with Nano silica: Figure 2 represents the flowchart of preparation of Nano silica magnetic composite materials using a micro twin screw extruder and micro-injection moulding equipment (make- Thermo Fisher, Germany). The Nano silica composites were made in several processes, beginning with weighing the components using a micro balancing system (Mettler Toledo, Switzerland) to determine their weight percentage ratio. After that, combined samples such as PVDF/Nano silica/$Ni_{0.8}K_{0.2}Fe_2O_4$ were well blended with the application of a kneader. The samples were then appropriately mixed and weighed. In polymer composites, the matrix and reinforcing materials are loaded in 90:10 ratios. Then, using a micro-injection molding machine, Nano silica composite materials were created, as illustrated in Figure 2. Maintain the kneader's rotor speed at 40-60 rpm while maintaining the temperature of the entire extruder unit at 300°C to 350°C for 20 minutes. The blended sample feed was then fed into a micro-injection plunger system with a pneumatic pressure of 60 bar once the necessary blending procedure was completed. Finally, a plunger was used to inject the extruded sample into a die, resulting in solid composite materials with the correct shape and size.

Figure 2: Flowchart for the process of production of Nano silica Composite.

3. Results and Discussion

3.1 X-Ray Diffraction of Nanosilica: It is reported that when the annealing temperature exceeds the temperature of 700°C, then the crystalline structure of Nano silica is obtained [27-28]. Generally, silica as amorphous structure available in rice husk. In this present research, we have used 1000°C to prepare the crystalline structure. Such results were also reported by some research groups [29-30]. The XRD spectra of the prepared Nano silica are shown in Figure 3.1 (a), and analysis was performed by a Bruker X-ray diffractometer with copper Kα radiation of wavelength 1.5405 Å. The plot was investigated in the range of angles 10°-90° with a scanning rate of 0.02°/minute. The peaks obtained in XRD analysis are which show that the nanomaterial prepared by RHA is crystalline and the crystal obtained is tetragonal. The prominent peak is observed at about 22 degrees, which confirms the formation of Nano silica material [COD 98015-3866]. The XRD plot is shown in Figure 3.1(a), having characteristics hkl value also indexed in the figure, which agrees with earlier reported work. [31-32].

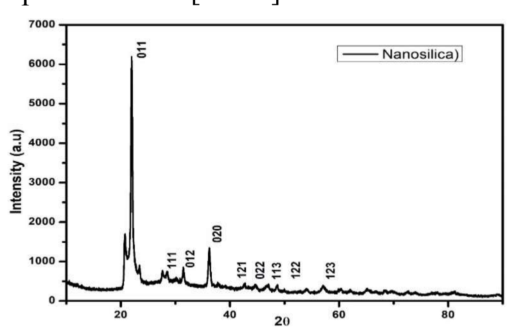


Figure 3.1(a): XRD spectrum of crystalline nano silica annealed at 1000°C

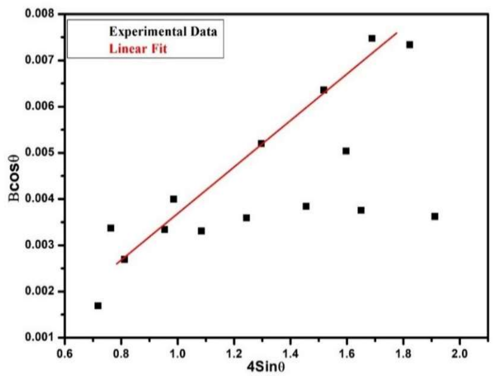


Figure 3.1 (b): W-H plot of Nano silica.

Further particle size was evaluated using Scherer's formula [33], which is discussed below:

$$D = 0.94\lambda/\beta \mathrm{Cos}\theta$$

Where D- average crystalline size of the particle, λ- wavelength of X-ray, β1/2- full width at half maximum, θ-Bragg's diffraction angle. The crystallite size was found to be 60.31 nm. Further, for more precise measurement, Williamson-Hall (Fig 3.1(b)) plot was used to evaluate crystallite size and strain. The result value found to agree with structural analysis, i.e, 62.11 nm and corresponding strain $2.84\text{x}10^{-3,}$ shown in Figure 3.1 (b). The present XRD measurement shows that the crystalline form of Nano Silica material from agricultural waste rice husk can be obtained at 1000°C using a low-cost chemical method.

3.2 Surface analysis measurement using Scanning Electron Microscope (SEM): SEM images are shown in Figure 3.2 (a), where rice husk nanoparticles are agglomerated of Nano-crystallinity and showing porosity. Thus, prepared nanomaterials can be a possible aspirant in electronics devices as these properties might be as a result of porosity and defects created due to vacancies created in the material [34-35]. Further, for a more precise measure histogram plot as shown in Figure 3.2(b) was analyzed, and the particle size was calculated and it was found to be 0.048 μm. Such porous structure functional materials from waste agricultural rice husk may be another novelty of this research.

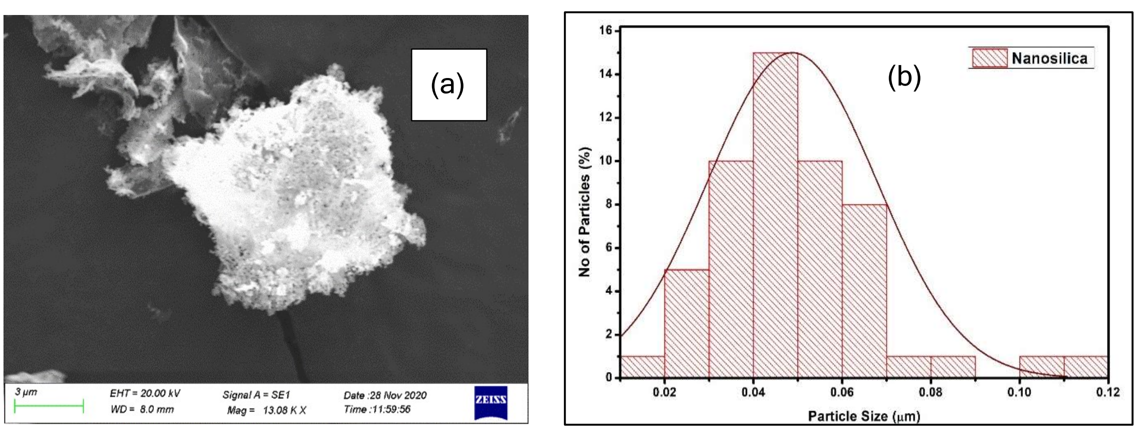


Figure 3.2 (a-b): SEM image and histogram plot of Nano silica nanomaterial obtained from waste rice husk.

3.3 Fourier Transform Infra-Red Spectrometer (FTIR): The FTIR spectrum of the prepared samples is shown in Figure 3.3. FTIR results show that the absorption peak at 3400- 3500 cm^{-1} confirms the presence of silanol–OH bond and H_2O. The other absorption occurring at 1095 cm-1 is due to the presence of i.e., Si-O-Si bond. The presence of Si-OH and other silane bond stretching vibrations. The peaks between 600-800 cm^{-1} are attributed to the stretching vibrations of C-H and C-Cl bonds. The FTIR graph confirms the presence of silica and is similar to the FTIR of Nano silica obtained in different works [36-37]. Thus, this measurement also agrees with the structural confirmation of the formation of Nano silica material. The presence of such functional groups in the material is responsible for various functional properties in industries.

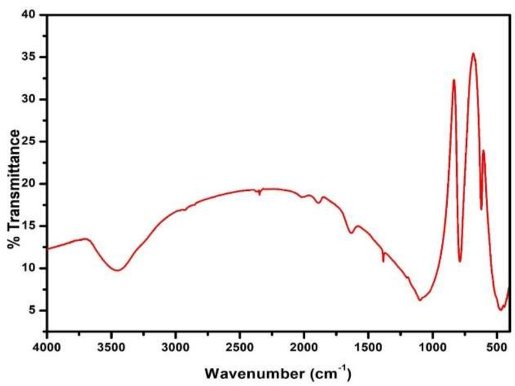


Figure 3.3: FTIR Spectra of Nano silica prepared at 1000°C for 2 hrs

3.4 Optical measurement and band gap calculation: For measuring the UV-Visible absorption spectra of crystalline Nano silica powder at normal incoming light in the wavelength ranges of 200-800 nm, a UV-Visible-NIR (Perkin Elmer spectrometer, UK) was used. The absorption spectra of the produced Nano SiO_2 sample are shown in Figure 3.4. The synthesized SiO_2 sample's absorption spectra display a larger absorption peak in the UV region, indicating that it can be used to boost catalytic activity. The visible area of the UV-Visible spectrum has low absorption, whereas the UV section has higher absorbance. The significant peak of the crystalline Nano silica sample is 350 nm, which is similar to a previous result by Ruchi Nandanwar et al [38]. The Tauc plots, shown in Figure 3.4(b-c), were also utilized to estimate the band gap of the nano-structured silica, synthesized using cost cost-effective leaching method. The indirect band gap was determined to be 1.73 eV, and subsequently direct band gap is 1.88 eV, which is lower compared to other semiconductor electronics materials prepared chemically by other research groups [39]. The difference is that prepared materials are obtained by using low-cost waste agriculture rice husk. The resulting band gap value is lower than that reported by Ruchi Nandanwar et al [40], which may be attributed to the 1000 °C high annealing temperature.

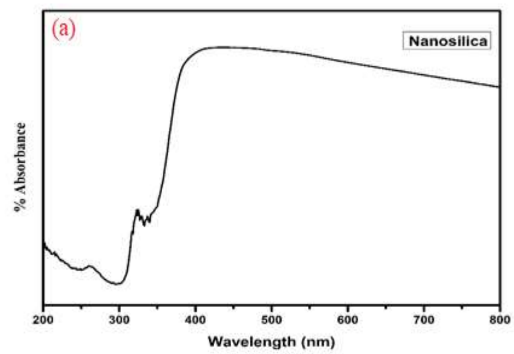
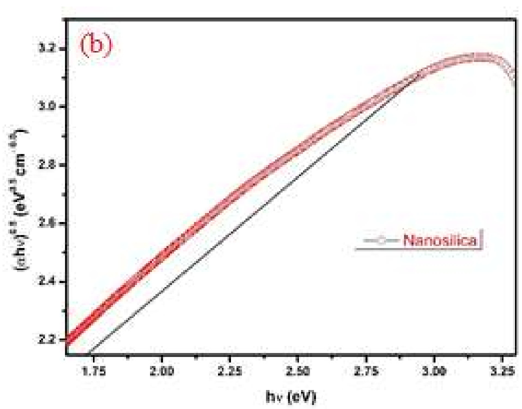


Figure 3.4 (a): UV-Visible spectra of crystalline Nano silica

3.5. XRD of Ferrite and Nanocomposite: The XRD pattern of $Ni_{0.8}K_{0.2}Fe_2O_4$, having been annealed at the low temperature of 500ºC for 3 hours, is shown in Figure 3.5 a. It has the entire characteristic peak reported by other research groups as well [41] and a crystallite size of 27.3 nm. Further, in the above section, the XRD pattern of crystalline Nano silica. In this section, we have shown the XRD pattern of the nanocomposite in Figure 3.5 (b). The pattern clearly shows the α and β-phases of PVDF nanoparticles at 18.5°, 19.8°, and 26.7°, having corresponding hkl values



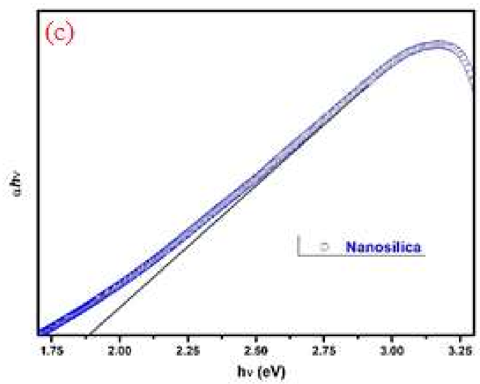


Figure 3.4 (b-c) Direct and indirect band gap of crystalline Nano silica

indexed in the plot. Further, other research groups also found that as the concentration of polymer is more it suppresses the other pattern. But it is clearly shown that the presence of SiO_2 and Ferrite nanoparticles is indexed in the XRD pattern. Further Williamson-Hall plots (shown in Figure 3.5 (c)) were done to investigate the crystallite size and lattice strain produced in the nanocomposite, and it was found to be 34.57 nm and 3.24×10^{-3}. The formation of Nano Silica composite decreases the crystalline size and strain as compared to pure Nano Silica.

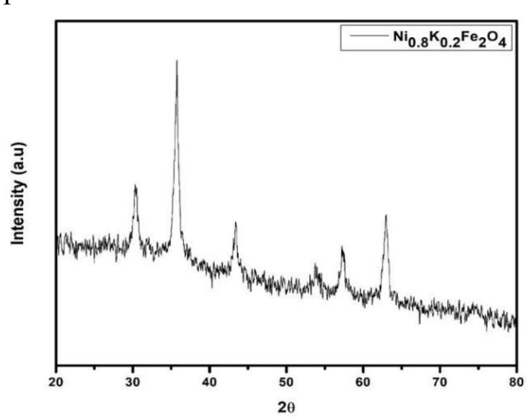


Figure 3.5 (a): XRD pattern of $Ni_{0.8}K_{0.2}Fe_2O_4$ nanoparticles.

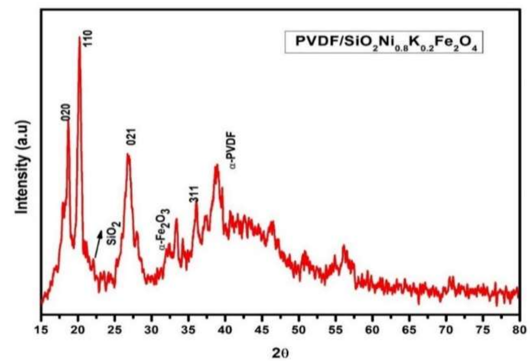


Figure 3.5 (b): XRD pattern of nanocomposite.

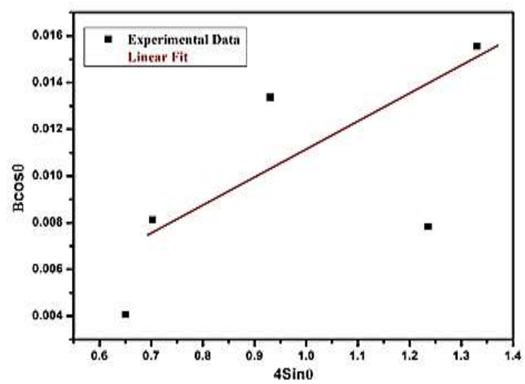


Figure 3.5 (c): Williamson Hall plot of the PVD/SiO_2/$Ni_{0.8}K_{0.2}Fe_2O_4$ nanocomposite.

3.6 Magnetic Measurement using Vibrating Sample Magnetometer (VSM)

The magnetic measurement was analyzed using Lakeshore 7400 in the applied field range of -10 KOe, +10 KOe, and the resultant graph is shown in Figure 3.6 (a-b). Nano silica is diamagnetic, as reported [42]. It is interesting to note that with the formation of PVDF silica and $Ni_{0.8}K_{0.2}Fe_2O_4$, it was noticed that the composite material shows ferrimagnetic properties, having a very low saturation magnetization of 0.0089 emu/gm and a coercivity value of 97 Oe. This magnetic parameter value is very less in comparison to its parent material, whose saturation magnetization and coercivity values are in the range of 18.95 emu/gm and 121 Oe. This sort of change might be a result of the suppression of the ferrite peak, as its concentration was low in comparison to other materials. So, this material can be used in flexible magnetic materials in semiconductor Electronics or in magnetic polymer [43-45]. Recently functional properties silica from rice husk have been used in drug delivery [46].

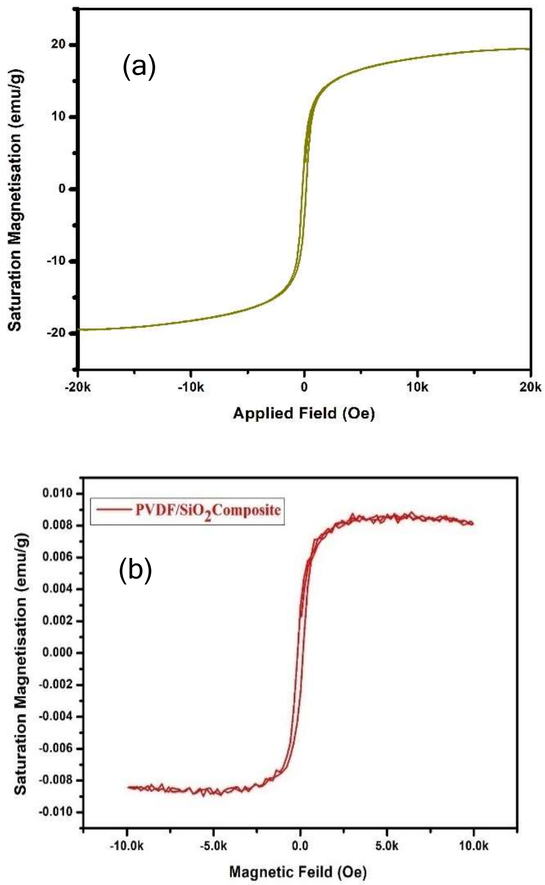


Figure 3.6 (a-b): Magnetic measurement of $Ni_{0.8}K_{0.2}FeO_4$ and $PVDF/SiO_2$ composite

4. Conclusions

In the current work, an economical and environmentally beneficial technology have been used to prepare crystalline nanosilica from rice husks, an agricultural waste. The tiny crystallite size is revealed by the XRD investigation. FTIR confirmed the presence bond nature of Si-O-Si, which is very important functional for its multifunctional properties. UV-visible have been used to investigate the optical properties of crystalline nanosilica, and the results show that it has outstanding optical properties in the visible range. It was discovered that the optical energy band gap was 1.78 eV, which is marginally lower than its bulk value and shows in semiconductor properties. UV–Vis spectroscopy showed strong UV absorption, indicating potential catalytic applications, and low visible light absorption. The formation of this nanocomposite using micro injection molding equipment was substantiated through XRD analysis, which displayed characteristic peaks of the composite constituents, indicating successful integration. Magnetic characterization revealed that the nanocomposite exhibited ferrimagnetic behavior, as evidenced by the presence of magnetic hysteresis loops. Magnetic property suggests that the nanosilica/ferrite/PVDF composites have potential applications as polymer magnets, offering a novel approach to developing magnetic materials from agricultural waste derivatives. This study demonstrates a sustainable pathway for synthesizing crystalline nanosilica from waste rice husk and its subsequent utilization in fabricating magnetic polymer nanocomposites, highlighting the potential of agricultural waste in advanced material applications.

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