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Synthesis of α-Fe_2O_3 Dispersed Polyvinyl Alcohol Nanocomposite Film

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α-Fe_2O_3 was purified from commercially available red oxide (IS-445). This purified α-Fe_2O_3 was dispersed in polyvinyl alcohol having m.w. 1,25,000 (obtained commercially by M/s Himedia Chemicals, Mumbai) through solvent casting method. The structure of synthesized composite film was studied by X-ray diffraction pattern (XRD) and morphology was studied by scanning electron micrograph (SEM). The variations in vibrational frequencies were studied by infrared studies.

Key Words: α-Fe_2O_3, Polyvinyl alcohol, Structure, Morphology, SEM, XRD, IR.

INTRODUCTION

Polymer nanocomposites are materials in which nanoscopic inorganic particles, typically 10-100 Å in at least one dimension, are dispersed in an organic polymer matrix in order to dramatically improve the performance properties of the polymer[1-3]. A system in which the inorganic particles are the individual layers of a lamellar compound, most typically a smectite clay or nanocomposites of a polymer (such as nylon) embedded among layers of silicates are produced, exhibits dramatically altered physical properties of the polymer.

It is expected that reducing the added particle size down to nanometric scale could enhance the performance of these materials, even though not at the wondering level as layer addition. These new materials are aimed at the substitution of more expensive technical parts and the production of barrier plastic film in food industry. Technical components, like gear systems in wood drilling machines, wear resistance materials, as well as films for food packaging, can be made out of these new materials.

Nanocomposite materials based on nanosized magnetic materials have been of great interest to researchers due to their possible applications in refrigeration and high-density information storage[4-6]. These composites are often prepared by dispersing magnetic materials in a non-magnetic matrix.

The present study represents the purification of α-Fe_2O_3 from commercially available red oxide and also emphasises upon the synthesis of α-Fe_2O_3 dispersed polyvinyl alcohol nanocomposite film. XRD, SEM, optical photography and IR studies characterize this film.

EXPERIMENTAL

Red oxide (IS-445), hydrochloric acid, liquid ammonia, polyvinyl alcohol of m.w. 1,25,000 and oxygen-free double distilled water are used. All the chemicals used are of AR grade. The method employed for the synthesis of polymer composite is solvent casting method.

Purification of α-Fe_2O_3

The purification of α-Fe_2O_3 from red oxide is reported in our earlier paper[7].

Minimum amount of red oxide (commercially available IS-445 grade) was dissolved in hydrochloric acid and was then reprecipitated by adding liquid ammonia (1 : 1) to the mixture; the precipitate was filtered, washed, and dried. The precipitated compound was transferred to a clean crucible and heated in an electrical oven until a red coloured powder was obtained (temperature maintained at 600°C).

Preparation of α-Fe_2O_3 dispersed polyvinyl alcohol composite film

The method employed for the synthesis of ferrite-polymer composite is reported in our earlier work[7]. A known weight of polyvinyl alcohol was dissolved in double distilled water and the required amount of α-Fe_2O_3 (5 wt. %) was added and stirred well in a magnetic hot plate. The α-Fe_2O_3 dispersed polyvinyl alcohol was transferred to a clean petridish and kept in a vacuum desiccator for complete evaporation of water. A red-coloured sheet-like film was obtaincd (hereafter referred to as PVA-α-Fe_2O_3). The prepared film is free from air bubbles and uniformly dispersed α-Fe_2O_3 particles.

Charactcrization

The powder X-ray diffraction patterns were obtained from GEOL JDX-8P or SEIMEN (Japan) X-ray diffractometer using CoK_α radiation and the indexing was done by employing PROSZKI program. The morphology of the ceramics was obtained from Leica Cambridge-440 scanning electron microscope. The infrared spectra of nanoceramics were recorded on a Perkin-Elmer FTIR spectrophotometer [Model 1000] in the range 4000–300 cm^{-1}.

RESULTS AND DISCUSSION

X-ray diffraction studies

The structure of pure polyvinyl alcohol and that of composited with α-Fe_2O_3 are studied by employing X-ray diffraction study.

Fig. 1 shows the XRD pattern of pure polyvinyl alcohol. There are no highly intense peaks throughout the spectrum. This indicates the amorphous nature of polyvinyl alcohol.

Fig. 2 shows the XRD pattern of PVA-α-Fe_2O_3 composite film. Sharp and highly intense peaks are observed. Some additional sharp peaks are also observed. d-Spacing values and the observed intensities of pure polyvinyl alcohol and its composites with α-Fe_2O_3 are listed in Table-1. An observation of this table indicates the presence of α-Fe_2O_3 in the polymer matrix.

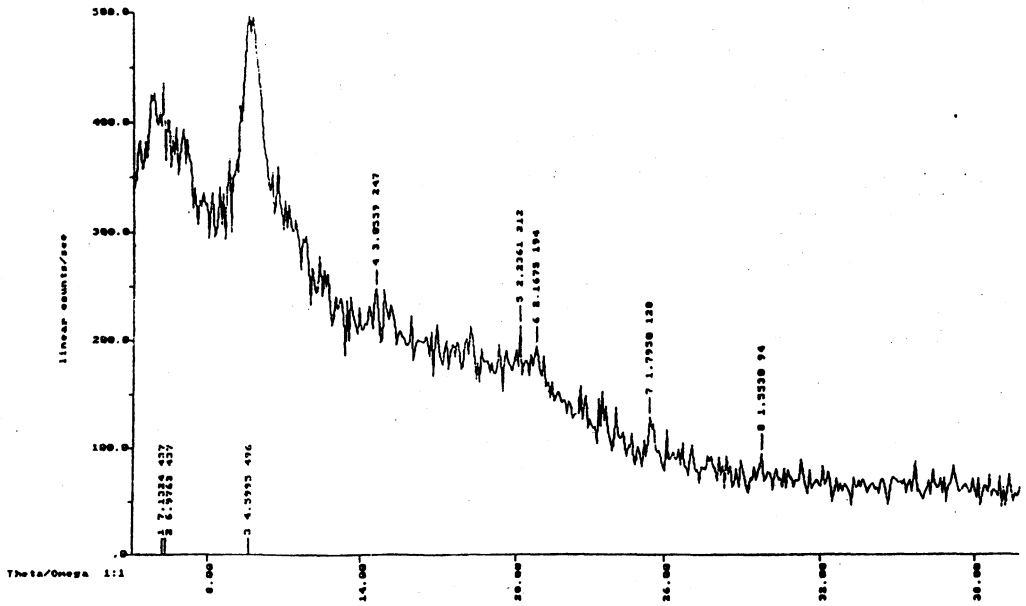

Fig. 1. XRD pattern of polyvinyl alcohol

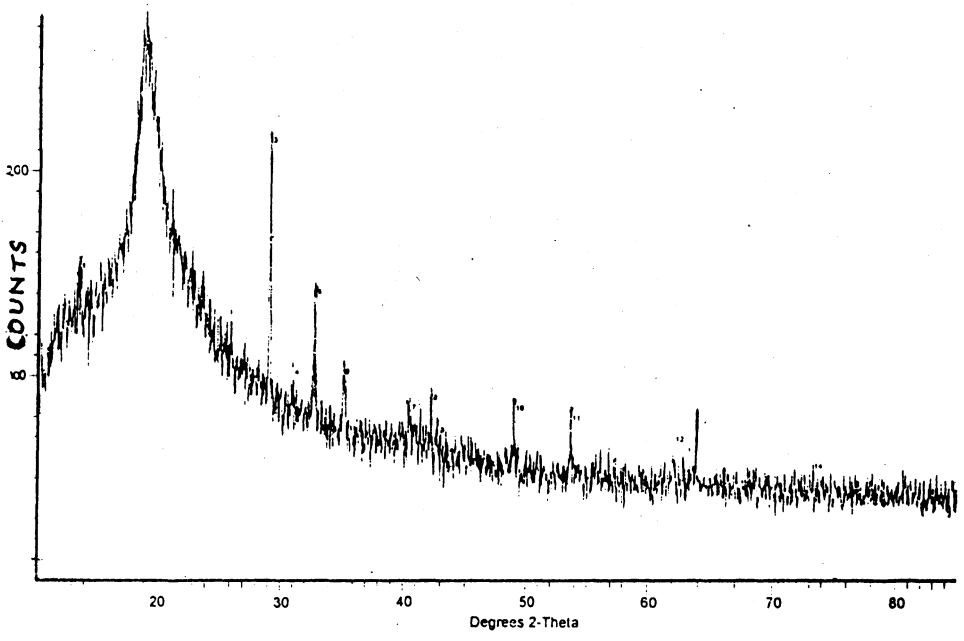

Fig. 2. XRD pattern of PVA-α-Fe_2O_3

Scanning Electron Micrograph

The surface morphology of pure polyvinyl alcohol and PVA-α-Fe2O3 composite was studied by scanning electron micrograph.

Figs. 3 (a) and 3 (b) show the SEM images of pure polyvinyl alcohol at low and high magnification.

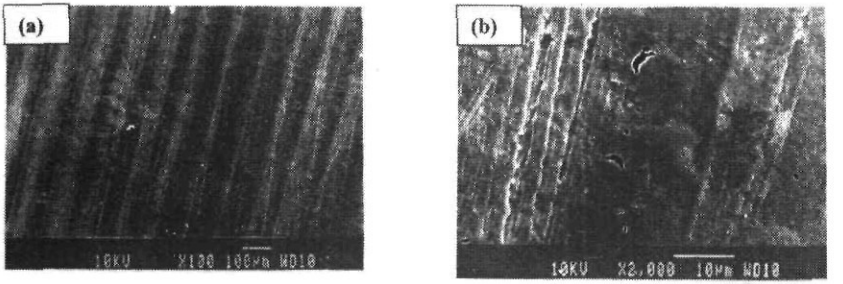

Fig. 3. (a) SEM of polyvinyl alcohol at low magnification
(b) SEM of polyvinyl alcohol at high magnification

The polyvinyl alcohol film is uniformly processed with streaks of uniform dimension and thickness. However, on higher magnification, we see that the film is not completely uniform but is somewhat disjointed and the separation between streaks is an amorphous sheet like form.

Figs. 4(a) and 4(b) show the SEM images of polyvinyl alcohol-α-Fe_2O_3 composite film at low and high magnifications. A clear disruption of the streak-like structure of polyvinyl alcohol is noticed. The polymer matrix now looks as a sheet with particles (either simple or sometimes agglomerates) uniformly distributed on the surface of the polymer sheet. Hence from this image it can be understood that the incorporation of α-Fe_2O_3 particles has changed the polymer morphology.

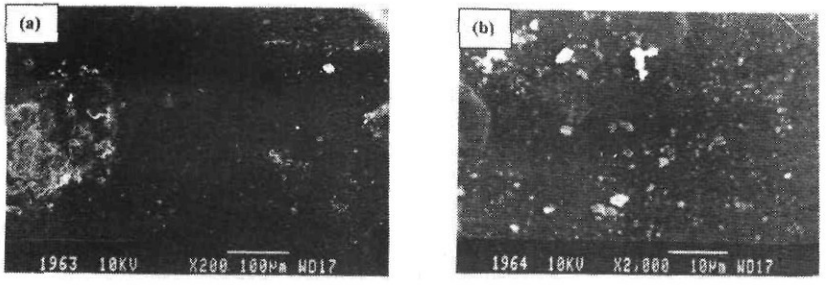

Fig. 4. (a) SEM of PVA-α-Fe_2O_3 at low magnification
(b) SEM of PVA-α-Fe_2O_3 at low magnification

Infrared studies

The vibrational frequencies of pure and comoposite polyvinyl alcohol were studied by infrared studies. Fig. 5 shows the IR spectra of pure polyvinyl alcohol. The vibrational bands are observed at 3190, 2170 and 475cm^{-1}.

Fig. 6 shows the IR spectra of PVA- α-Fe_2O_3 composite film. Some additional peaks from 1700 cm^{-1} to 900 cm^{-1} are observed. The vibrational frequencies of pure polyvinyl alcohol and PVA-α-Fe_2O_3 composite film are given in Table-2. These additional peaks are due to dispersion of α-Fe_2O_3 particles into the polymer matrix.

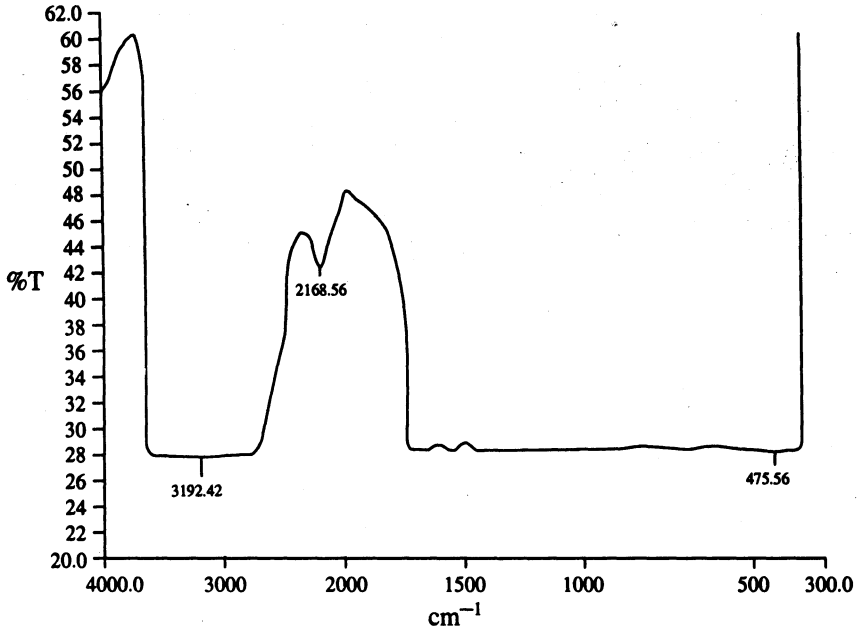

Fig. 5. IR spectra of polyvinyl alcohol

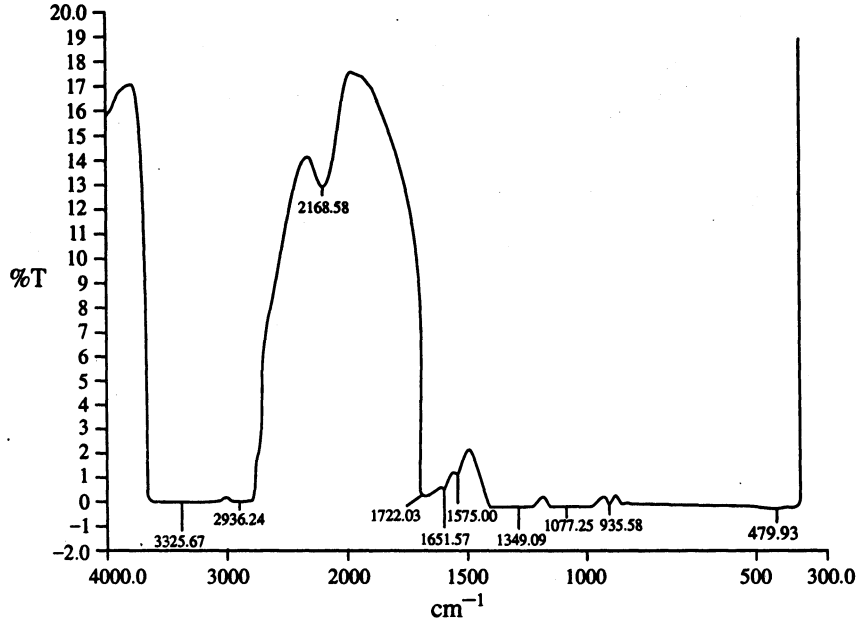

Fig. 6. IR spectra of PVA-α-Fe_2O_3 composite film

TABLE-1
d-SPACING VALUES OF PURE POLYVINYL ALCOHOL AND ITS COMPOSITE WITH α-Fe_2O_3

S.No.	d-Spacing values of pure polyvinyl alcohol	Intensity observed for polyvinyl alcohol	d-Spacing values of α-Fe_2O_3-PVA composite film	Intensity observed for composite
1.	4.5995	496	6.627	52
2.	3.0559	247	4.710	100
3.	2.2361	212	3.083	74
4.	2.1675	194	2.894	34
5.	1.7950	120	2.740	48
6.	1.5530	94	2.561	34
7.	—	—	2.229	27
8.	—	—	2.143	29
9.	—	—	2.116	23
10.	—	—	2.186	27
11.	—	—	1.711	26
12.	—	—	1.497	22
13.	—	—	1.459	25
14.	—	—	1.293	17

TABLE-2
VIBRATIONAL FREQUENCIES OF PURE POLYVINYL ALCOHOL AND ITS COMPOSITE WITH α-Fe_2O_3

S.No.	Vibrational frequencies of pure polyvinyl alcohol	Vibrational frequencies of α-Fe_2O_3-PVA composite film
1.	3190	3325
2.	2170	2936
3.	475	2168
4.	—	1722
5.	—	1651
6.	—	1575
7.	—	1350
8.	—	1078
9.	—	935
10.	—	480

Conclusions

1. The distribution of α-Fe_2O_3 particles is uniform throughout the polymer matrix.
2. The crystallinity is developed due to addition of α-Fe_2O_3 in to the polymer matrix
3. There is a possibility of forming α-Fe_2O_3 composite with other polymers.

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