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Chromium Doped Neodymium Oxide PVA Nanocomposites: Electrical and Optical Revelations

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***Abstract*— The present work emphasis on the effect of Chromium doped neodymium oxide nanoparticles on optical, electrical, morphological and micro crystalline properties of resultant PVA nano composite. PVA/NdCrO3 were fabricated using solvent casting method with varying concentration of neodymium chromium oxide nanoparticle viz (0.0, 1.0, 2.0, and 4.0wt./wt.%). The obtained nano composites are subjected to X-ray diffraction analysis (XRD), Scanning Electron Microscope (SEM) to evaluate their phase behaviour and morphological properties of nanocomposites. UV-visible Spectroscopy, Photoluminescence Spectroscopy, were employed to evaluate their optical properties. LCR meter was used to examine the electrical properties of prepared nanocomposites. The UV–visible spectra reveals that there is decrease in indirect band gap of PVA from 4.98 to 2.46 eV and direct band gap from 3.9 to 1.08 eV compared to pristine PVA. NdCrO3 doped nanocomposite which shows multi-fold enhancement in optical characteristics such as refractive index of nanocomposites compared pristine PVA, optical conductivity, extinction coefficient, Urbach's energy on increasing in dopant concentration. The electrical properties such as dielectric constant (ε′), dielectric loss (ε″), and electric modulus (M′ and M″) of the nanocomposite were measured over a range of frequencies. The dielectric constant decreases with increase in frequency and the contents of nanofiller in PVA matrix. Whereas, dielectric loss increases with frequency and decreases with nanofiller contents. From the obtained results it is clear that the obtained nanocomposites open a new window as multiple materials.**

***Key words*— PVA, Nanocomposites, Polymer matrix, Optical properties, $NdCrO_3$.**

I. INTRODUCTION

The increasing applications of highly flexible, biodegradable, hydrophilic optoelectronic materials have opened the technological window towards the design and development of newer flexible matrices with novel material properties [1]. The inorganic/organic nano structured matrix are being potentially visualized as advanced multifunctional systems in optical wave guides, transparent conductive coatings, solar cells for photovoltaic conversions and so forth [2]. PVA is generally used as transparent optical systems due to their optical clarity and enhanced flexibilities and narrow refractive indices [3] limiting their optical applications. However, PVA as dielectric materials is known as a particle stabilizer that offer excellent processability in addition to filler dependent property turnabilities [4]. The polar PVA with high dielectric constant is an excellent host that offers significant filler stabilities leading to highly homogeneous composite systems [5], with the –OH pendant groups of PVA assisting uniform filler dispersions.

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The incorporation of the nanoparticles into polar polymers can induce significant changes in the properties of polymers [6]. Furthermore, the high surface reactivity attributed to large surface-volume ratio of nanofiller intercalations may induce totally new optoelectronic properties to the polymeric matrix [7].
In addition, among the lanthanide series, neodymium oxide (Nd2O3) is regarded as the most important rare earth oxide. For the fabrication of solid-state lasers, crystals of the light, glasses such as welding goggles and sunglasses Nd2O3 was employed due to the greyish blue compound and dichroic properties [10]. Nd2O3 is used for sophisticated applications including magnetic devices and protective coatings because of its desirable property [8]. Because of its high dielectric constant, Nd2O3 is essentially electrically and thermally stable. In addition, its conduction band is well suited for use in microelectronics [9]. These features make Nd2O3 a suitable material for optical applications and other semiconductor devices. Furthermore, Cr2O3 because of its unique thermodynamic stability, hardness, chemical resistance, and antiferromagnetic qualities, Cr2O3 stands out among metal oxides. [11] In the past, several researchers have investigated a range of applications based on Cr2O3 sNPs, such as sensors, photocatalysis, solar cells, fuel cells, piezoelectric devices, green pigment and protective coating, and catalysis, have been accomplished [12]. Various research group have investigated electrical and optical properties of Nd2O3 and Cr2O3 nanocomposites separately. In the present study, an attempt is made to suitably integrate varied weight fractions of Chromium doped neodymium nano fillers into a polar PVA to establish the effect of dopant content on the electronic band structure and hence, revelation of opto-electronic properties of nanocomposite (NC).

II. Experimental Section

A. Materials and methods

AR grade material Neodymium nitrate [Nd(NO3)3], Chromium nitrate [Cr(NO3)3] were procured from sigma Aldrich, whereas, glycine [C2H5NO2] from S.D fine chemicals were used to prepare NdCrO3 nanomaterials. All the chemicals were used without further purification and all the solutions were made with distilled water. Solution combustion and solution casting method was used for preparing NdCrO3 and its PVA nanocomposites respectively.

B. Characterization

The photonic absorption and transmission characteristics have been established by UV–visible spectrophotometer, Schimadzu-1800 (Japan) in the wave length range 190-1100nm. The electrical properties of the PVA composite films were measured according to the ASTM standard using a high-frequency LCR meter, Wayne Kerr 6500 P (UK) with suitable electrodes (50 mm in diameter and 0.12 mm thick) between two circular electrodes. The success of PVA/NdCrO3 NC formation was ascertained from their microstructural and morphological examinations, using an X-ray powder diffractometer (XRD) PROTO AXRD Benchtop (Canada) and Scanning Electron Microscope (SEM), Zeiss-108A (Germany). The elemental composition of nanomaterial was analysed using EDX.

C. Synthesis of Neodymium chromium oxide nanoparticles

To synthesize NdCrO3 nanoparticle, oxidizer is prepared by adding stoichiometric amount of Nd(NO3)3 and Cr(NO3)3 in a clean beaker and 15ml of distilled water was added and stirred under magnetic stirrer for 25 mins. Stoichiometric amount of Glycine is used as fuel and dissolved in distilled water on constant stirring. Both oxidizer and fuel were mixed with continuous stirring for 30 minutes under alkali pH. The mixture was further stirred under elevated temperature until the gel is obtained. It is then transferred into clean silica crucible and was calcinated in a muffler furnace at 600 ¬oC for 5 hrs. After grinding by using a pestle and mortar. Golden yellow colour fine powder of NdCrO3 nanoparticle was obtained.

Fig: 1. NdCrO3 nanoparticle synthesized using solution.

D. Fabrication of NdCrO3/PVA nanocomposites

NdCrO3/PVA NCs was prepared by environment friendly solution intercalation method. Initially 7.5 wt% of PVA solution was prepared (mol wt≈ 84000) by continious stirring for 90 mins at 65 °C. 0.5, 1.0, 2.0, and 4.0 wt% of NdCrO3/PVA NCs was prepared by adding calculated amount of nanofillers to the above prepared PVA solution. Then this solution is stirred for 5 mins, followed by sonicating for 10 mins to achieve uniform distribution of the nanofillers in the polymer matrix. Prepared solution is then drop casted on clean glass mould and the solvent is allowed to evaporate in room temperature. After complete evaporation of the solvent the films were removed from the mould and used for the further analysis.

III. RESULT AND DISCUSSIONS

A. XRD Analysis

Structural properties of the PVA matrix due to addition of nanofiller is studied using X-ray diffractometer. Fig. 2a. shows the X-ray diffraction patterns of NdCrO3 NPs. It can be seen that many sharp peaks were observed in the X-ray profile, which can be attributed for the presence of the crystalline nature of the synthesized NdCrO3 nanoparticles. The peaks at 12.7, 29.2, 31.03, 39.66, and 54.12 are correspond to planes present in NdCrO3 nanoparticle. Fig. 1b. shows the peaks due to crystalline planes present in nanocomposites. An intense characterstic peak of pristine PVA can be observed at 2θ=19.9̊ as shown in the Fig. 2b. which corrosponds to a diffraction plane 111 and indicates the semi-amorphous nature of PVA [13]. The Scherrer lengths (L) of the PVA and its nanocomposites have been calculated using Scherrer formula [14]

$$L = \frac{K\lambda}{\beta cos\theta} \qquad (1)$$

where, k is the constant (k=0.9), which is related to the crystallite shape, λ and θ are the radiation wavelength and Bragg's angle, respectively; and β is the full witdth at half maximum of the diffraction peak. The variation in the intensity of relative diffraction peak is an indicator that shows the intraction between the polymer and the added nanofiller. From the figure 2b it also clear that the increase in filler content the intensities and the shape of the peak corresponds monotonic intraction between the filler and the polymer host [15]. The diffractograms of PVA and its nanocomposites shows concentration dependent nature as the intensity decreases with increase in nanofiller concentartion which is attributed to the increased H-bond interaction between PVA chains and NdCrO3 at the interface [16]. Which also responsible leads to variation in the interplaner distance (d-spacing) shown in Table 1.

Further more, the lattice strain (β cos θ/4) and dislocation density (η=1/L1/2) have been calculated for the peak at 2θ=19.9o for all nanocomposites. The obtained lattice strain and dislocation density increases with an increase in the dosage of NdCrO3 nanoparticle in the PVA matrix, which leads to increase in structural disorder in PVA matrix. Thus, amorphous nature of nanocomposites increases with increasing nanofillers in PVA matrix. Further, the micro-structural parameters such as inter-crystallite separation (R) and stacking fault (SF) were determined using the formula [17].

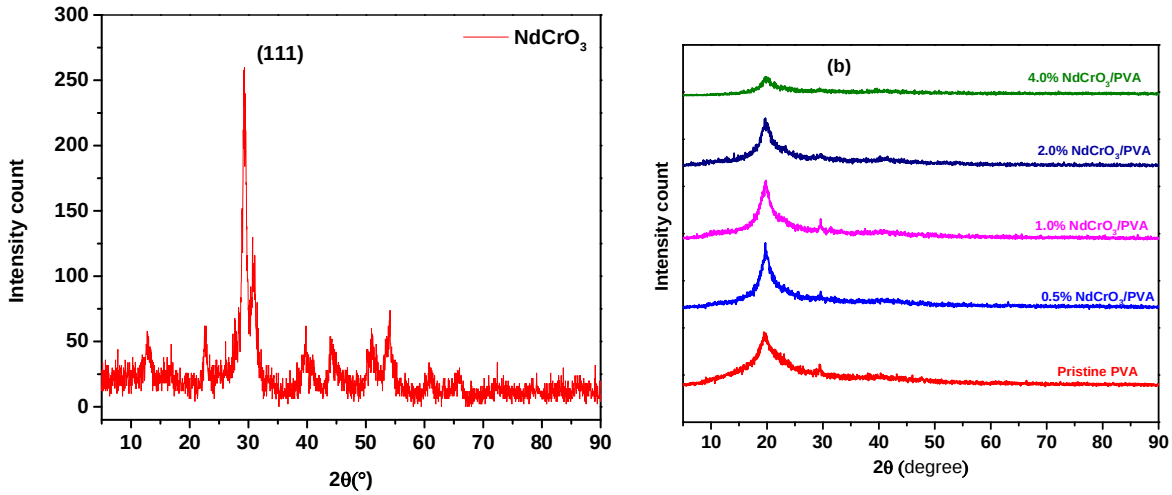

Fig: 2. a) XRD pattern of NdCrO3, b) XRD pattern of NdCrO3/PVA nanocomposites

TABLE I: THE STRUCTURAL PARAMETERS OF PVA/NDCRO3 AT CORRESPONDING TO PVA 2Θ MAX

$NdCrO_3$ /PVA (Wt/wt%)	$2\theta_{max}$ (˚)	(β)	L (nm)	SF	ηx 10^19	g	d_{hkl} (nm)x 10	Lattice strain
Pristine PVA	19.44	1.40	0.10	4.03	0.98	1.45	2.5	1.06
0.5	19.72	0.96	0.15	1.81	4.13	2.06	1.78	0.74
1.0	19.84	1.08	0.13	1.87	5.22	2.14	1.78	0.84
2.0	19.72	1.16	0.13	2.03	5.65	2.32	1.58	0.91
4.0	19.76	1.25	0.12	2.24	6.86	2.55	1.71	0.97

$$R = \frac{5\lambda}{8sin\theta}, \quad SF = \left[\frac{2\pi^2}{45(tan\theta)^{\frac{1}{2}}}\right]\beta \qquad (2)$$

Where, λ is the wavelength of X-ray (1.54 Å), θ is the diffraction angle, and β is the FWHM of prominent intensity peak. From Table 1. the variation of R with increased SF values are attributed to the physical interaction of polymer chains with incorporated NdCrO3 nanoparticles. The XRD results may be referred to an increase in the amorphous nature of PVA where similar kind of result was reported in the earlier literature [18].

B. SEM (Scanning Electron Microscopy)

Surface morphology, degree of dispersion, and distribution of nanoparticles in the PVA matrix is examined by using SEM. The SEM photomicrographs of NdCrO3 PVA and its various composites are illustrated in Figure 4(a-e). The NdCrO3 NPs exhibit plate-like structure as shown in Fig 3. The pristine PVA shows the uniform and plain surface morphology and the NdCrO3 are well dispersed in the PVA matrix as shown in Fig 2 (b-f) respectively. The nanoparticles are well dispersed in PVA matrix enhancing the physical connection between the matrix and filler interface. With an increase in filler concentration in NCs it exhibits agglomeration forming the nanoclusters in the nanocomposite.

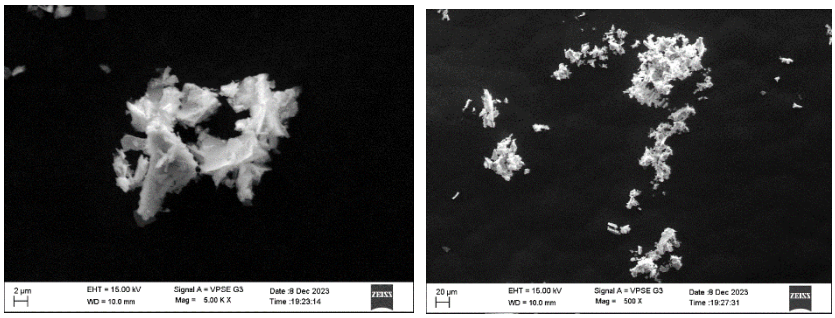

Fig 3. SEM images of NdCrO3 Nanoparticle

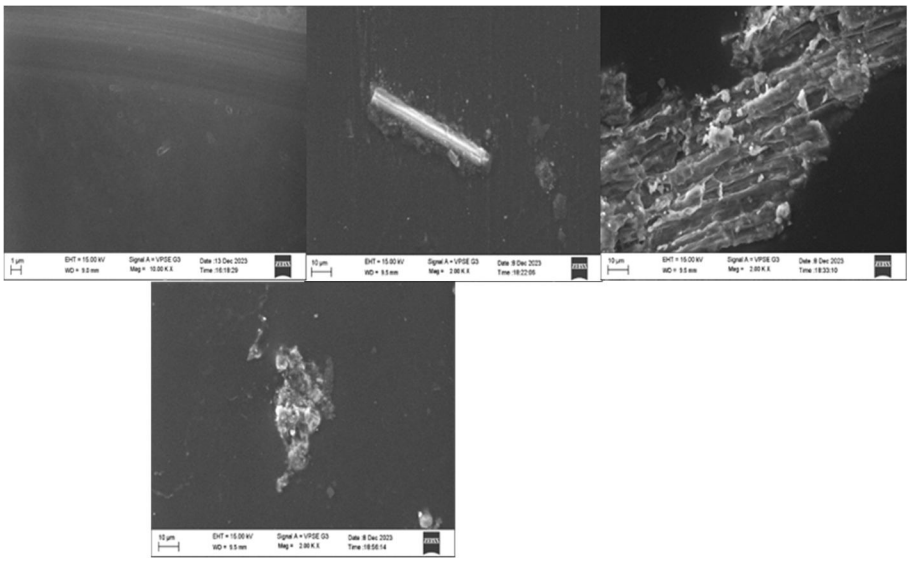

Fig 4. SEM microphotograph of a) Pristine PVA, b) 0.5%, c) 1.0%, d) 2.0% and e) 4.0% wt/wt% of NdCrO3 in PVA

The energy dispersive spectrum (EDS) illustrated in Figure 5, shows the atomic and weight % of the individual elemental composition of NdCrO3, nanoparticles where the obtained results in Table 2. indicate the NdCrO3 NPs contain Nd, Cr, O and fabricated nanocomposites have Nd, Cr, C and O elements which indicates that all the elements are present in the fabricated nanocomposites and the absence of other elemental impurity.

C. UV-vis studies

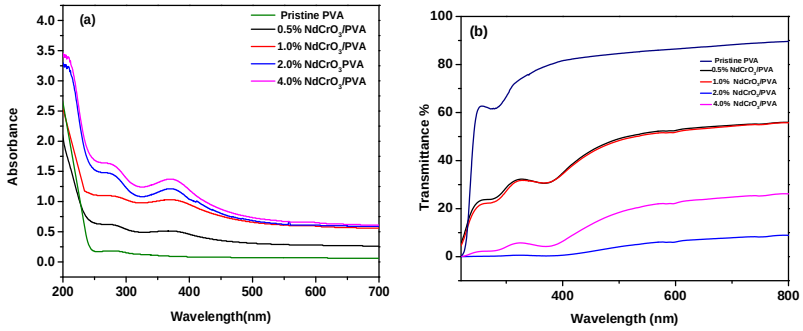

Fig: 5. (a) UV–visible absorbance of $NdCrO_3$/PVA NCs,(b) Transmittance of $NdCrO_3$/PVA NCs.

To study optical properties of $NdCrO_3$/PVA nanocomposite, UV spectra was employed, absorbance and transmittance were recorded. The absorbance spectra recorded in the UV–visible region (200–700 nm) is depicted in Fig. 5a. PVA exhibit characteristic broad-shouldered valley in the wavelength range 260–280 nm, which falls under UVC region but on increasing the filler content the broad shouldered valley shifts to UVB and UV C region. Absorption in this region can be ascribed to $\pi \rightarrow \pi^*$ electronic transition arises from unsaturated C=O residual acetate groups in PVA structure and C=C which is present in the tail head of the polymer [19]. While the new peak appearing at 350-380 nm validates the presence of $NdCrO_3$ nanofillers, these peaks are assigned to $n \rightarrow \pi^*$ inter band transitions for free electrons of oxygen atoms in polymer chain. From figure we can observe that the new peak at 350 to 380nm is red shifted with increasing in nanofiller dosage with increasing in absorption intensity. The transmittance data shows that with increasing in nanofiller dosage transmittance decrease which is in good agreement with previously reported journals [20]. Hence this type of nanocomposites can be used as UV blockers.

Evaluation of absorption edge using absorption coefficient

Absorption coefficient is measure of how far light can travel through material before it gets observed. It is given by formula,

$$\alpha = 2.303(A/t) \qquad (1)$$

where, $\boldsymbol{\alpha}$ is absorption coefficient and A is absorbance, and t is the thickness of the film. The graph of absorption coefficient vs energy is plotted in Fig 6. From the Table2 absorption edge of pristine PVA is around 5.4 eV, while for PVA/$NdCrO_3$ nanocomposite, their positions were found to be displaced towards lower energy values exhibiting a minimum of 1.15eV for 4 wt. % of PVA/$NdCrO_3$ nanocomposite film. The shift in absorption edge towards lower energy or higher wavelength may be attributed to the effective dispersion of nanofiller in the PVA matrix. Also, the increase in absorption coefficient with an increase in weight percentage of nanofillers may be attributed to the absorption by $NdCrO_3$. [21]

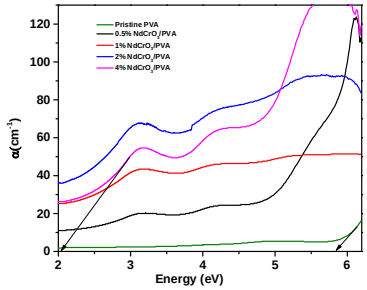

Fig: 6. Absorption coefficient of NdCrO3/PVA as a function of photon energy

TABLE: II. EFFECT OF NANO NDCRO3 CONTENT ON THE OPTICAL CONSTANTS OF PVA FILMS

$NdCrO_3$/PVA (wt/wts%)	Absorption edge (eV)	Direct energy band gap	Indirect energy band gap	Urbach's energy E_u(eV)
0.0	5.7	5.40	5.30	2.0
0.5	2.2	1.9	4.72	1.5
1.0	2.0	1.8	3.56	1.6
2.0	1.38	1.6	3.02	1.4
4.0	1.8	1.5	2.73	1.2

Optical energy band gap of NdCrO3/PVA nanocomposites

PVA nanocomposites are excellent materials for solar cell encapsulants due to their barrier and flexible properties hence optical energy band gap is a key factor which decides which portion of sunlight to be absorbed. [22] The optical band gap of a material is the energy difference between the valence and conduction bands (CB) it explains optical transitions occurring in solid systems. Smaller the band gap, higher will be its charge conduction. [23] From Tauc's plot the optical energy band gap can be determined.

$$(\alpha h\nu)^{1/m} = B\,(h\nu - E_g) \qquad (2)$$

where α is the absorption coefficient, hν is the photon energy, E_g is the optical energy band gap, B is proportionality constant, m is used to determine the type of electronic transition. For direct transition m is equal to ½ and for indirect transition m is equal to 2. The graph in Fig 8 a and b shows direct and indirect band allowed transitions.

Table 3. implicates that optical band gap is directly dependent on nanofiller content, with energies allied to inter-band electronic transitions showing a steady decrease from (5.4 eV) for pristine PVA to (1.5 eV) for 4 wt% of PVA/$NdCrO_3$ NC film. The decrease in optical energy gap revels a change in the optical band structure of PVA films upon nanofiller intercalations due to formation of polarons in the $NdCrO_3$ nanofiller introduced PVA NCs [24]. Moreover, the $NdCrO_3$ nano fillers might make it possible for lower energy transitions to occur, which would improve photoconduction, by creating a conducting channel between the gaps in the highest occupied and lowest unoccupied molecular orbitals (HOMO–LUMO). [25] The increase in structural randomness resulting from the modifications in polymeric structure post nano filler doping is also reflected in the decrease of optical band gaps. [26]

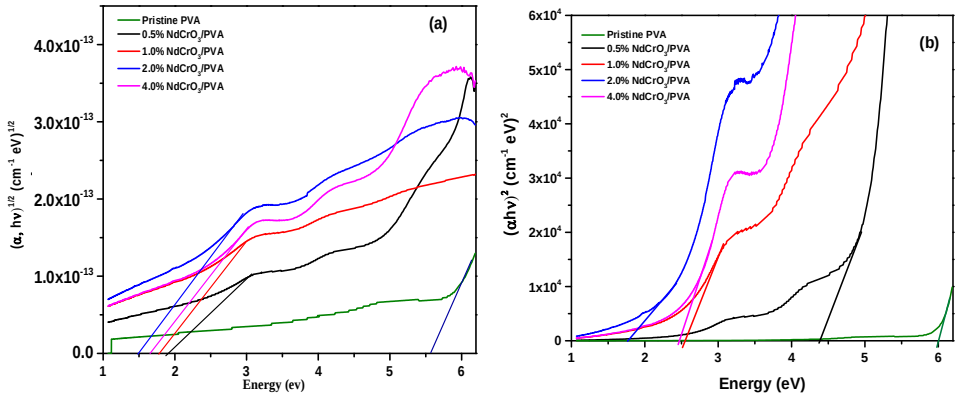

Fig: 7. a) The plot of $(\alpha h\nu)^{1/2}$ direct energy, and b) $(\alpha h\nu)^2$ indirect energy as function Photon energy for varying contents of nanofiller

D. Electrical property

Dielectric constant

The dielectric constant (ε′) is a measure of the ability of a material to store electrical energy. The dielectric behaviour of a polymer is based on the charge distribution and statistical thermal motion of its polar groups and also physical structure has a great influence on the same [27]. Dielectric properties in polar materials are dependent on dipole attachment to the main chain and segmental mobility. The dielectric constant of polar molecules is high and affected by temperature, composition and frequency variations [28]. Dielectric properties are a complex function of bulk permittivity, conductivity, size, shape, spatial arrangement of the constituents (the filler and the matrix), and the frequency [29]. Polycrystalline character of PVA creates a substantial amount of disorder, the dielectric properties of which can be improved by addition of metal oxide nanoparticles. The dielectric parameter as a function of frequency is described by the complex permittivity in the form;

$$\varepsilon'(\omega) = \frac{C.t}{\varepsilon.A} \qquad (3)$$

Dielectric constant of $NdCrO_3$/PVA as a function of percentage composition and frequency is show in the above figures 8. The initial high dielectric constant at low frequencies in polar materials like PVA maybe from electrode and interface influences on the sample, causing ε′ to decrease as the frequency of the AC field rises [30].

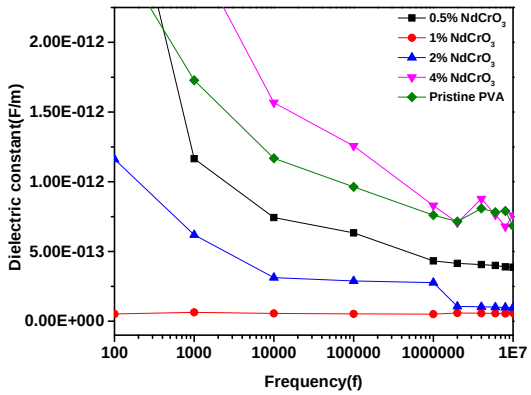

Fig: 8. Plot of Dielectric constant vs frequency

Dielectric loss

Dielectric loss refers to the dissipation of energy in a dielectric material when it is subjected to an alternating electric field. This loss typically occurs due to factors such as dipole reorientation and molecular friction within the material, leading to a conversion of electrical energy into heat. ε″ is always greater than zero and is usually much less than ε′. The variation of ε″ as a function of frequency for PVA/NdCrO3 composite films with different NdCrO3 concentrations is shown below.

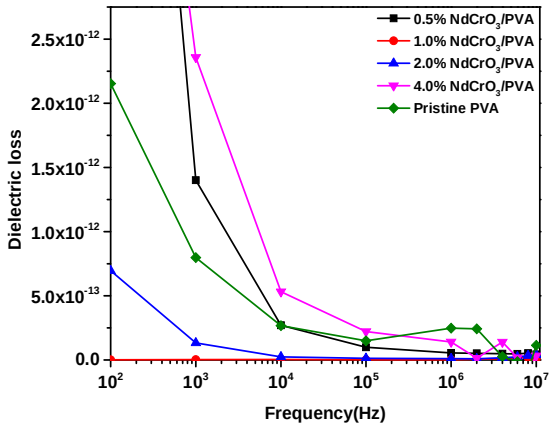

Fig: 9. Dielectric loss of $NdCrO_3$/PVA nanocomposite vs frequency

It is clear that as frequency increases, ε″ lowers maybe because of mobile charges within the polymer backbone. At higher concentrations of $NdCrO_3$ nanoparticles, higher values of ε″ at low frequency can be explained in terms of electrical conductivity. Free ions originating from $NdCrO_3$ nanoparticles increase with concentration and decrease ε″ at a higher frequency [31]. Furthermore, ε″ is significantly influenced by the hydroxyl groups present in PVA [32].

Electric modulus

The electric modulus physically corresponds to the relaxation of the electric field in the material, so that the electric modulus represents the real dielectric relaxation process. Electric modulus is defined as the inverse quantity of complex permittivity. Employed in order to analyse the dielectric response of PVA/$NdCrO_3$ nanocomposite films also helps in interpretation of bulk relaxation phenomena in complex systems [31].

$$M' = \frac{\varepsilon'}{(\varepsilon')+(\varepsilon'')} \quad (4)$$

The plots of the electric modulus (M') as a function of filler loading and frequency for PVA and PVA/$NdCrO_3$ nanocomposites containing different weight percentage of filler shown in the graphs below.

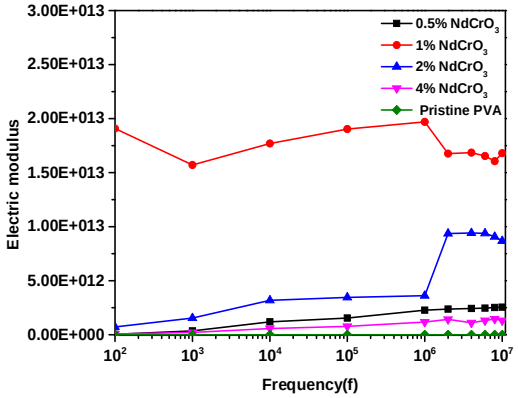

Fig: 10. Electrical modulus (M) of $NdCrO_3$/PVA nanocomposite as function frequency.

Due to the suppression of the high capacitance phenomenon in M' and M'', the primary benefit of permittivity and conductivity relaxation treatments is that space charge effects frequently do not obscure the features of the spectra.

E. Photoluminescence analysis (PL)

Fig: 16. shows PL spectra of $NdCrO_3$/PVA nanocomposites with emission monitoring at 300nm. All samples appear to have had broad visible light emission spectra in the 410–500 nm region, with two peaks associated with emissions at 420 and 475 nm, respectively. The presence of oxygen vacancies and inherent defects, which are the source of free carriers, is widely recognized as the primary cause of visible light emission in oxide nanomaterials.[33]. Thus, origination of the first and second peaks is due to the transition resulting from a recombination of electron-hole pairs between oxygen vacancies and metal ions [34]. The transition for the first and most intense peaks located at 420 nm produced photons of violet light, while for the second and less intense peak located at 475 nm photons of blue light were produced.

F. Atomic force microscopy Analysis

Morphological studies were performed by Atomic Force Microscopy and change in surface morphology with addition of NP as shown in Fig. 11(a,b, c, d). The various parameters like variation of Average roughness, RMS roughness and Average height of PVA and its nanocomposites with various concentrations of $NdCrO_3$ nanoparticles in PVA are calculated from AFM data (Table). RMS roughness of PVA increases with addition of 0.5, 1.0, 2.0 and 4.0% concentration of NPs.. This could be due to more nucleation sites produced by NP. The increase in RMS roughness due to agglomeration of NP

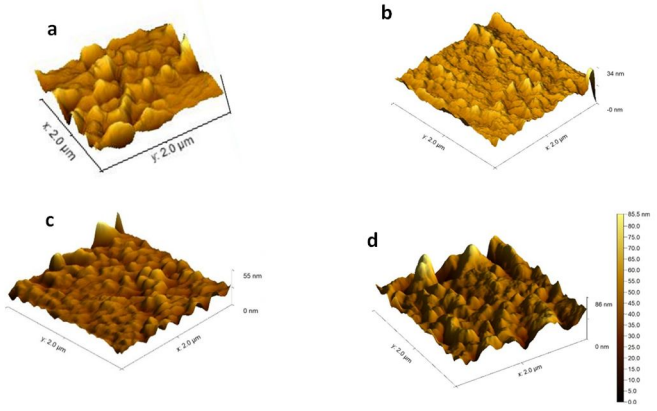

Fig: 11. The following images shows AFM images obtained by tapping method

IV. CONCLUSION

The Chromium Doped Neodymium Oxide PVA nanocomposites have been synthesised using the solution combustion technique for various concentrations (0.5,1, 2, 4 wt%) using the solution casting method. The present study aimed to examine how various characterization methodologies are impacted on $NdCrO_3$ nanocomposites. The XRD analyses show that semi-crystalline PVA- NdCrO3 composites have formed and the $NdCrO_3$ is well dispersed throughout the PVA matrix and the SEM images confirm the agglomeration in the nanocomposite, which also validates the better dispersion of nanoparticles in the PVA matrix. We studied the optical properties of $NdCrO_3$ nanocomposites where, the UV–visible absorption studies show an absorption in the range of 250 to 350nm and for both direct and indirect allowed transition, the optical band gap of $NdCrO_3$ nanocomposites at different concentrations (0,0.5,1,2, and 4 w%) drops from 5.40 eV to 1.5 eV and 5.30 to 2.73 eV. So, this could be a promising material in optoelectronic device applications. Based on all of the aforementioned optical studies, it was found that adding $NdCrO_3$ to the PVA matrix increases both its optical conductivity and the absorption properties.

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