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STUDY OF STRUCTURAL, MICROSTRUCTURAL AND THERMAL PROPERTIES OF RARE EARTH OXIDE DOPED POLYANILINE

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Abstract

Yttrium oxide doped polyaniline were successfully prepared through the In-situ polymerization reaction method to a different percentage of weight in the presence of oxidizing agent ammonium dichromate. The composite was analysed by XRD, FTIR, morphologic structure was considered by FESEM, whereas thermal equilibrium by TGA and DSC. The resultant of FTIR, XRD showed that the composition of composite has changed substantially by the addition of Y_2O_3 , TGA-DSC analysis desirable that the thermal stability of yttrium oxide doped polyaniline was giant than that of polyaniline this indicates that the composites are gradually changing from amorphous state to crystalline state.

Keywords: Polyaniline, Ammonium dichromate, Yttrium oxide, FESEM.

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1 Introduction:

The growing attentiveness in nanocomposites conducting polymers are the latest field in enlarging technological and scientific interest, contribution of opportunity to synthesize an extensive variety of encouraging new materials [1]. Conducting polymers are used in newly discovered electronic device like, field effect transistors, supercapacitors, biosensors, light emitting diodes and the polymer materials are used for elastomers, surfactants etc. Miniaturization of equipment increasing their effectiveness and reducing costs. The need for new materials. The examine of conducting polymers showed that treat the semiconductor properties. They may exist in various oxidation states and together with inorganic semiconductors, they react to outside influences by changing certain characteristics like magnetic properties of conductivity, colour due to those conductive polymers are termed intelligent polymers. off all the conducting polymers, polyaniline (PANI) is included which not require any precautions or special equipment's for the synthesis. PANI was the first of the conductive polymers to be like electromagnetic material for batteries and condensers, good environmental stability, and unique doping/de-doping property. Antistatic coating, detecting material for sensors [2]. Most of the methodologies used for nanocomposite synthesis are usually based on two tracks as a redox step reaction in which simultaneously polymerisation of aniline and forming of nanoparticles take place, another one is In-situ polymerization where the pre-synthetic nanoparticles are missed in monomer solution followed by chemically or electrochemical polymerization [3] PANI is synthesised by chemical oxidative polymerization method. In this polymerisation monomer is subjected to a chain reaction with the forming of systematic macro-molecules and developmental chains at the same instant categorized as complex

supramolecular structures. Consequence, conductive polymers holding a steady supramolecular structure along with numerous morphologies is formed. The majority structures may not be dissolved or fused without demolition of polymer chain and modify properties of polymer. Consequently, synthesizing is the only method to control structure of polymer chain and acquire nanocomposites forms of the conducting polymer.

There are generally two methods for the synthesis of conducting polymers as electrochemical oxidation of the monomers or chemical oxidation of the monomers, specifically: Electrochemical Polymerization and chemical Polymerization, in electrochemical polymerization electrode potential is increased such that it overreaches to +1.05(RHE). In chemical synthesis of PANI, a large-scale of oxidizing agents are used having giant oxidation potentials exceeding +1.0V they are, persulfates, dichromate's, cerium (IV) salts etc. the distribution of polymer chain in acidic medium essential overthrow energy barrier equivalent has a potential for oxidation of +1.05V. when the dispensing starts, the oxidative potential of reaction diminishes. where as in chemical synthesis of PANI, persulfates possess oxidative potential of +2.01V are used broadly. Even so some of demonstration be revealed that aniline is also oxidized through the week oxidizer process with potential close to +1V. such agents have the potential to oxidize aniline in neutral or basic environment otherwise than, in acidic medium the potential oxidation barrier is high though the use of low oxidizing agents. beginning a few difficulties because wouldn't give necessary route to the forming of resultant products [4].

Polyaniline has trailed the substantial recognition due to its decisive moment, for the dawn of unpublished composites and their invitational investigations foremost to the abundant application of technological

importance. PANI has been studied widely with the metalloids, nonmetals, metals, and mainly with the inorganic metal oxides. These all have massive research interest and provided new designs to the transmission implement in these polymer composites. These composites with PANI establish vast collection of applications [5]. Even so PANI has some drawbacks, like low ductility, mechanical presentation, which restrict its application to control these difficulties, PANI is standardly constituted with other inorganic materials, [6-9] among prolific inorganic particulate types, metal oxides are in large part thermally stable, which exercise improve the thermal stability of PANI [10-17]. Rare earth oxide exhibits the welfare of its bio-degradable stable and machinable properties. In this awareness, PANI's combination with rare earth oxide establish an engrossing technique for enlarging next generation of driving organically-inorganic hybrid materials, from the combination of the recommendable properties of the inorganics, like heat resistance and mechanical strength with electronics properties of Polyaniline [18-20]. However, beyond there's a restricted study with rare earth oxide to amplify the study of Polyaniline with rare earth oxides I have been taken yttrium oxide as a doping material because now a day's rare earth oxide has become a hot research topic because of its broad spectrum of applications as good thermal stability, unique physical and chemical properties. These materials were used in many these include computer storage, rechargeable batteries, mobile telephones, fluorescent and illumination used in X-ray, MRI Scanning system. PANI and PANI-Y₂O₃ Composite with varying different wt% of Y₂O₃, was synthesized using in situ chemical polymerization in the presence of Ammonium Dichromat (ADC) as oxidizing agent. the thermal stability and structure, interaction between elements is analysed.

2 Experimental section:

2.1: Materials:

Aniline (C₆H₅NH₂), Ammonium Dichromat (NH₄)₂Cr₂O₇, Hydrochloric Acid (HCL), and Yttrium Oxide (Y₂O₃) all chemicals were taken S. D. Fine chemicals, During the entire synthesis of PANI and PANI-Y₂O₃ Composites, distilled water has been used.

2.2 Synthesis of Polyaniline:

Polyaniline was synthesized by chemical polymerization reaction. 0.25M of aniline and 1N of HCL were mixed together with stirring, to form the beginning compound aniline hydrochloride. 0.25M of ADC was mixed dropwise to the reactant composition with continuous stirring for 5h. at ambient temperature to undergo polymerization. The sample was kept overnight to settle down. Sample has been vacuumed filtered and washed multiple times with distilled water and 1N HCl to remove unreacted particles. The obtained final product was kept for drying to remove moisture and grinded the dried product to get uniform powder of pure polyaniline.

2.3 Synthesis of PANI-Y₂O₃ Composites:

PANI-Y₂O₃ was synthesized through In-situ chemical polymerization reaction method and same process was repeated for preparation of composite. Stoichiometric amount of yttrium oxide added before formation of final product with varying different wt% and kept it 5hrs for constant stirring at room temperature. After completion of polymerization sample was kept to settle precipitate at the bottom, then it was purified using vacuum technique and repeatedly washed with distilled water and 1N HCl to remove unreacted particles. The obtained product was kept for drying and dried sample was grinded to get PANI-Y₂O₃.

3. Results and Discussion:

3.1. XRD Analysis:

The XRD images of pure Polyaniline and Polyaniline doped Y_2O_3 composites are shown in Figure (1a). The pure PANI reveal wide peak at 2θ around the angle 26° which specify the PANI has amorphous in nature this peak can be assigned to the dissemination of PANI chain at interplanar spacing [21]. Figure (1b) manifest the diffraction spikes corresponding to single phase of cubic Y_2O_3 [JCPDS 41-1105] [22]. As Y_2O_3 content is enlarge in the composite, as 5%, 10%, 15%, 20% and 25% PANI's wide diffraction peaks become strong. A new peak appears at 2θ angles around 29° , 33° , and 48° when the amount of Y_2O_3 oxide is increases [23] and the diffraction peaks of Y_2O_3 this can be clearly seen in the composites, with a slightly varying of intensity, this indicates that nature of the PANI is changes from amorphous to crystalline by addition of

Y_2O_3 . Figure (1c, 1d, 1e, 1f, and 1g) reveals X-ray diffraction images of PANI- Y_2O_3 composites, in which most of the peaks of the Y_2O_3 have appeared in the composites, this confirms that Y_2O_3 holds its uniform structure after the formation of PANI- Y_2O_3 composites [21]. Additionally, the mean crystalline size in the composite was observed at about 71 nm as deliberated by Debye-Scherrer's formula given in equation (1). And applied on the strongest peak at $2\theta=29^\circ$.

$$t = \frac{k\lambda}{b\cos\theta} \dots \dots \dots (1)$$

In which, k is a constant regarded as 0.9

λ - wavelength of the X-rays used ($\lambda=1.541 \text{ \AA}$)

b- is the full width at half maximum of the most intense peak of XRD pattern

2θ - Bragg angle.

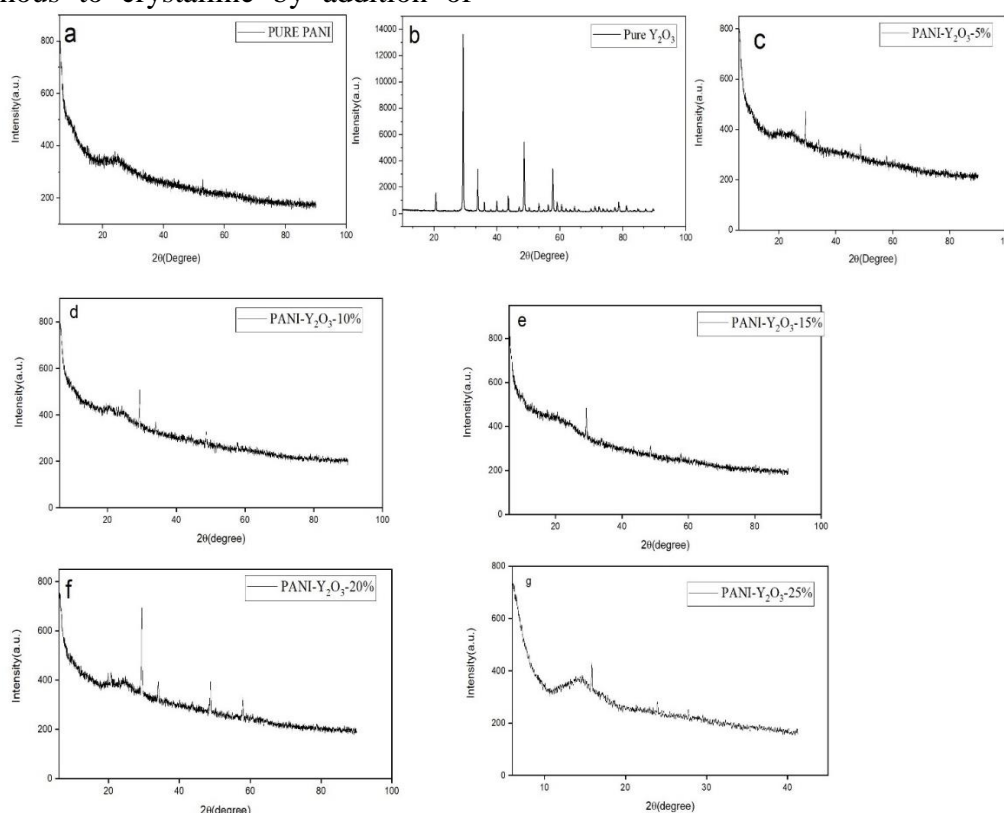


Fig 1: X-ray Diffraction pattern of

a) Pure PANI b) Pure Y_2O_3 c) PANI- Y_2O_3 -5% d) PANI- Y_2O_3 -10% e) PANI- Y_2O_3 -15% f) PANI- Y_2O_3 -20% g) PANI- Y_2O_3 -25% Composites

3.2: TGA Analysis:

TGA measurement were carry out to analyse the thermal stabilities of PANI and PANI-Y₂O₃ Composites. The TG curve of pure PANI, PANI-Y₂O₃ Composites with different amount of Y₂O₃ are shown in fig (2) [24]. Thermal data obtain by TGA delineate the developing and comprehensive stability of material upon decomposition [25]. Degradation temperature obtained from PANI and PANI-Y₂O₃ composites thermograms, the composites reveal a five-step pattern of weight loss that the first detectable weight losses was 3.2% and 2.3-3.7% at consequent temperature range of 29-270⁰C for pure PANI and any PANI-Y₂O₃ composite samples, respectively. The reason for this initial stage is a loss of moisture. The Second break-up was found at 270-420⁰C for PANI with a weight reduction of 1.39% and at 280-445⁰C for PANI-Y₂O₃ composites with weight loss 0.89-1.22%. This degradation stage looks like an expulsion of unresponsive monomer and free HCl [26]. In stage three for PANI the loss is 1.5% at 420-580⁰C and 1.03-1.35% for PANI-Y₂O₃ composites at 425-600⁰C. Mass loss has been detected in the

fourth stage to approximately at 580-900⁰C and the resultant value was 2.3% for PANI and 1.7-2.1% for PANI-Y₂O₃ composites such a step may be suitable for more breakdown of the PANI structure that required the highest temperature to decompose. The degradation pattern for the composite of the PANI-Y₂O₃ is similar to that of PANI exhibits a four-step process of mass loss when the quantity of Y₂O₃ is increases the curve shows a weight loss at five-stage of ambient temperature to 900⁰C in fifth step mass loss of PANI-Y₂O₃ composite is 1.5-1.9% at 790-900⁰C and the thermal stability of the composites was further enhanced [27]. Total PANI weight loss was reduced by 8.5% in temperature range 29-900⁰C [28] and PANI-Y₂O₃ composites has losing weight is as good as PANI with 8% [29]. It can be concluded from the above TGA thermogram it can be seen that thermal stability is greatly influence by an interaction between PANI and rare earth oxide like Y₂O₃. An appropriate amount of oxide Y₂O₃ make the composites more stable while more or less quantity than this decreases its thermal stability, thus the thermal stability of PANI could be directly controlled by selecting loading of a pertinent amount of Y₂O₃[30].

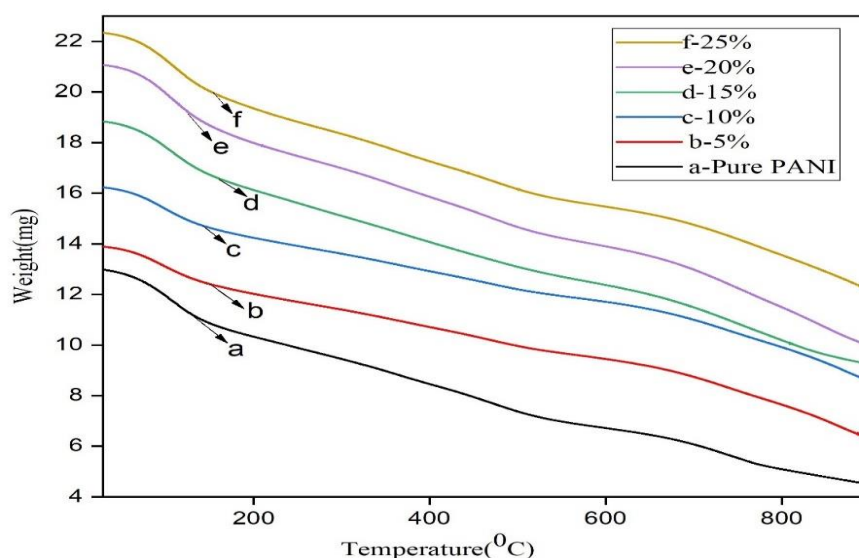


Fig 2: TGA plot of a) Pure PANI b) PANI-Y₂O₃-5% C) PANI-Y₂O₃-10% d) PANI-Y₂O₃-15% e) PANI-Y₂O₃-20% f) PANI-Y₂O₃-25% Composites

3.3 DSC Analysis:

The DSC curve of Polyaniline and PANI with different weight percentage of Y_2O_3 rare earth oxide is shown in given figure (3) and (4). It shows only one broad endothermic dip at $100.21^\circ C$ in the Pure PANI this indicates that the Pure PANI is in amorphous nature as shows in figure (3) and the first endothermic dip of PANI- Y_2O_3 Composites with 5,10,15,20 and 25 wt% of Y_2O_3 doping are observed at $102.56^\circ C$, $103.20^\circ C$, $109^\circ C$, $104^\circ C$ and $107.62^\circ C$ respectively in figure (4) [31]. This peak can be linked to the development of water as moisture absorbed by the polymer [32]. As it is also understandable from the figure

(4) that as doping percentage of composites are increases another one endothermic dip is appeared in composites as $513.48^\circ C$, $530.10^\circ C$, $507.03^\circ C$ and $526.05^\circ C$ by the addition of 5, 10, 20 and 25% respectively, were as in 15% composite of PANI- Y_2O_3 there will be two endothermic dip at $532.60^\circ C$ and $703.82^\circ C$. The increase in endothermic dip of composites is caused by the strong intermolecular interaction of Y_2O_3 particles with PANI, apart from this the uniform dispersion of Y_2O_3 particles expand. These interactions resist the mobility of polymer chain and hence increased an endothermic dip in composites [33]. This indicating that the sample was in amorphous state with little crystallization.

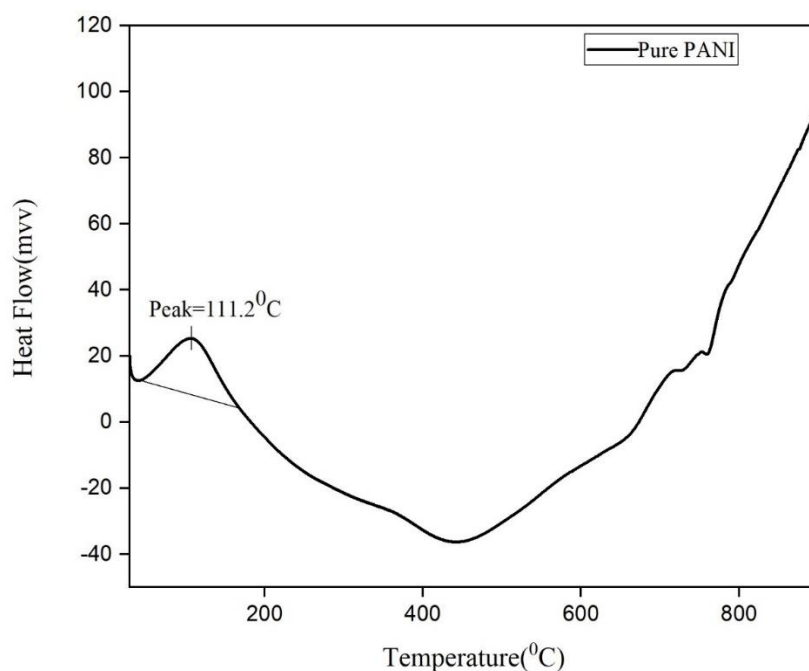


Fig 3: DSC cure of Pure PANI

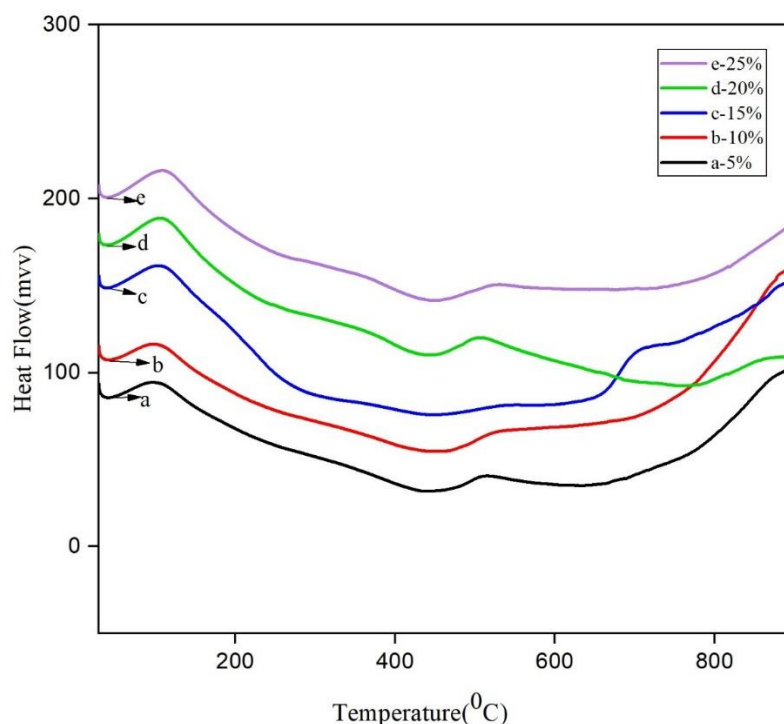


Fig 4: DSC curve of PANI-Y₂O₃ Composite's

a) PANI-Y₂O₃-5% b) PANI-Y₂O₃-10% c) PANI-Y₂O₃-15% d) PANI-Y₂O₃-20% e) PANI-Y₂O₃-25%

3.4: FTIR Analysis:

FTIR spectra registered for Pure PANI and PANI-Y₂O₃ composites are shown in figure (5) & (6) respectively. PANI's FTIR spectrum exhibits Characteristic peaks in 3300-3000 cm⁻¹ [19]. The absolute PANI peaks are distributed as follows 1578, 1500, 1341, 1312, 1253, 1150, 826 and 694 cm⁻¹. The peaks 1578 and 1500 cm⁻¹ are assigned to C=C bands of benzenoid and quinoid rings respectively [34]. the presence of these two bonds indicates that the polymer is composed of amine and imine unites [35]. The peaks at 1312 and 1253 cm⁻¹ are assigned to the C-N groove modes of the benzenoid rings, the peak at 1150 cm⁻¹ is attributed to a bending vibration in the interior plane of the C-H bonds formed during the protonation, the peak at 826 cm⁻¹ is assigned to the off-plane distortion of C-H in the p-dissociated benzene cycles. The

introduction of Y₂O₃ altered the conjugated backbone structure of PANI. The peaks of PANI-Y₂O₃ composites. These occurrences specify that the Y₂O₃ particles connected to the PANI chains and affected the absorption characteristics of the composite across them the interaction between PANI and the Y₂O₃ partials, as shown in fig (6) The corresponding peaks of pure PANI at 1578 went up to 1580 cm⁻¹, 1500 have been shifted to 1505 cm⁻¹, 1341 have risen to 1379 cm⁻¹, 1312 are now 1316 cm⁻¹, 1150 have increased to 1153 cm⁻¹, 826 went up to 828 cm⁻¹, 694 have been shifted to 695 cm⁻¹ wavenumbers in PANI-Y₂O₃-20% composites. These discrepancies clearly indicate the Vander Waal's type of interaction between PANI & Y₂O₃ particles during the composite formation [36].

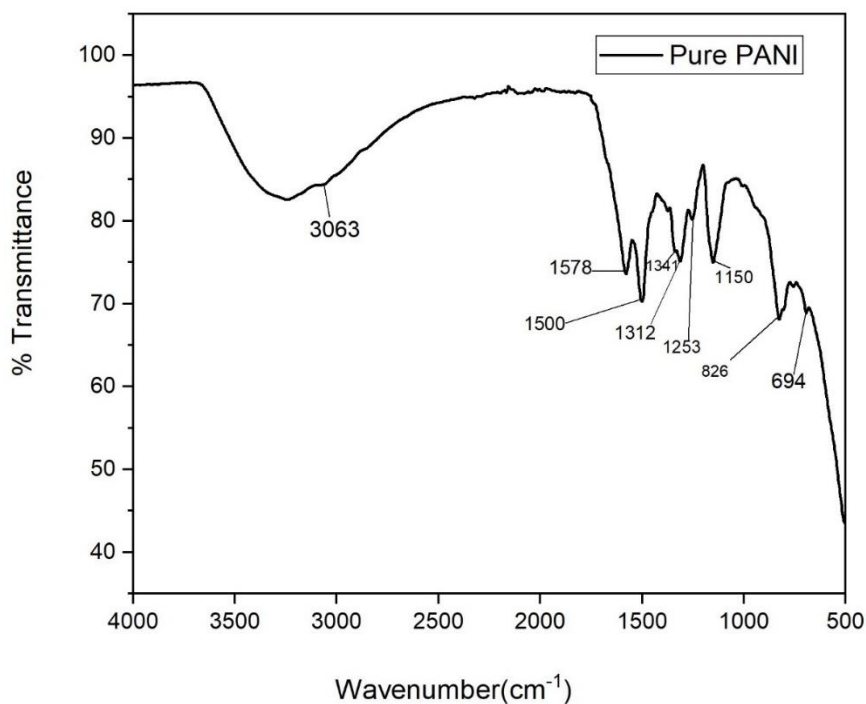


Fig 5: FTIR Spectra of Pure PANI

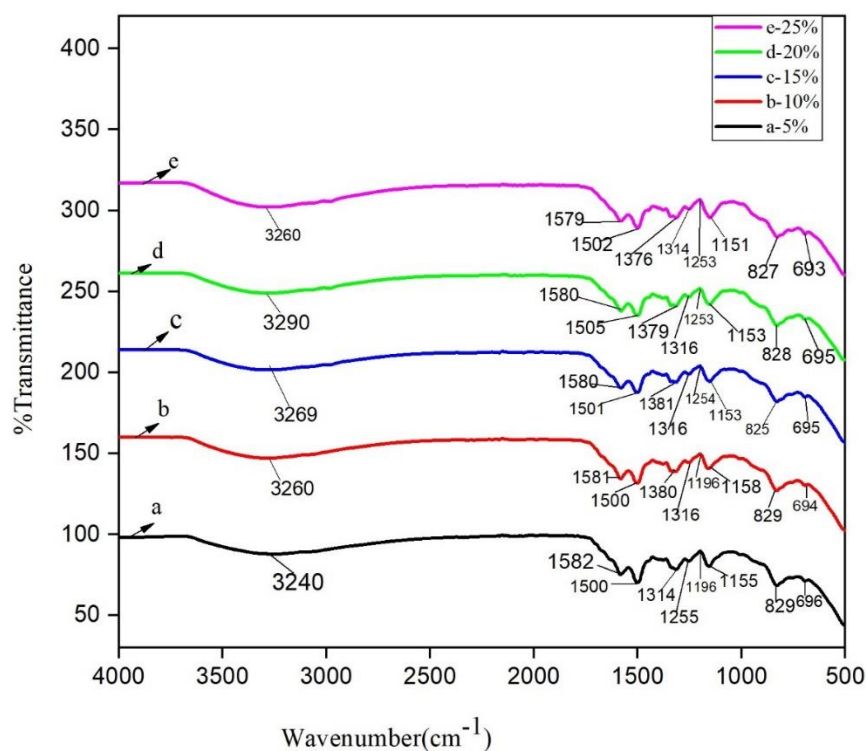


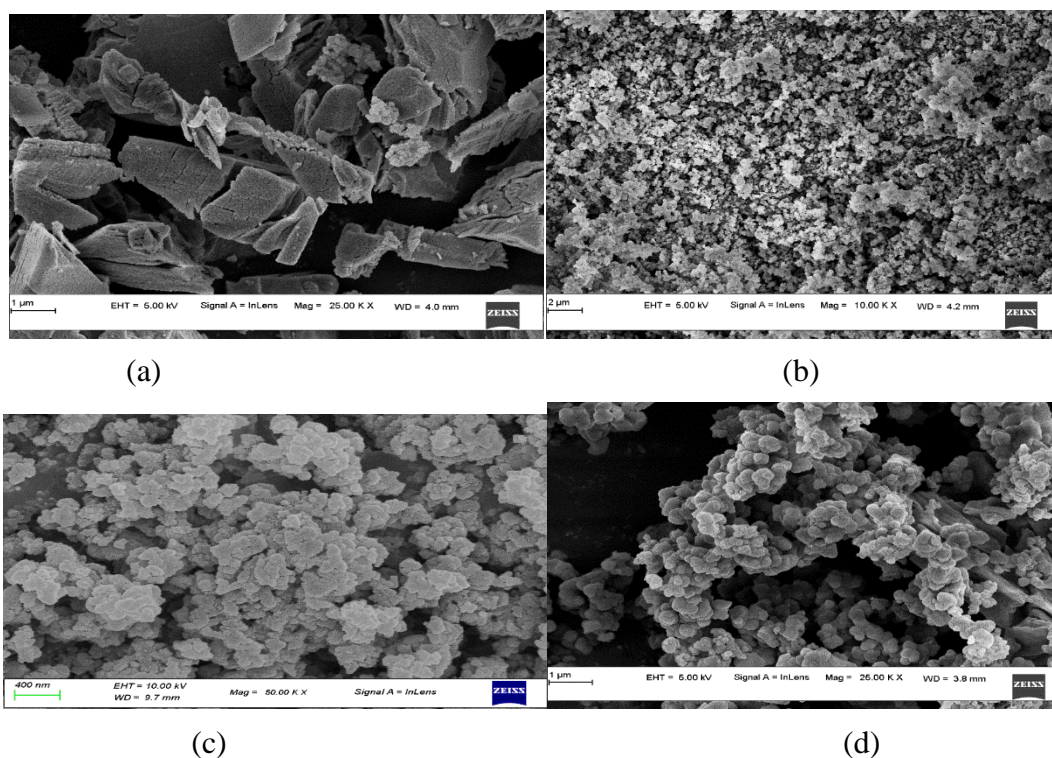
Fig 6: FTIR Spectra of PANI-Y₂O₃ Composite's

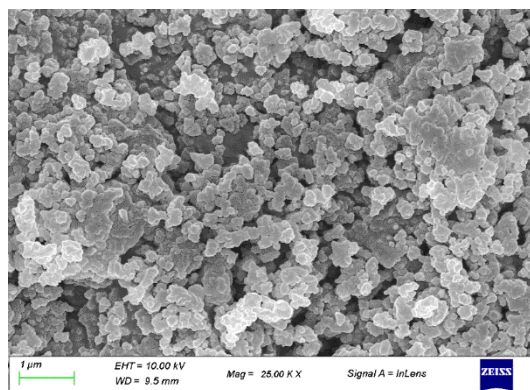
- a) PANI-Y₂O₃-5% b) PANI-Y₂O₃-10% c) PANI-Y₂O₃-15% d) PANI-Y₂O₃-20% e) PANI-Y₂O₃-25%

3.5: FESEM Analysis:

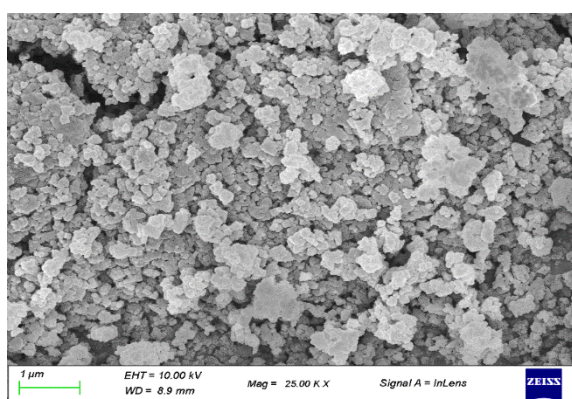
Figure (7) illustrates the FESEM and EDX images from these samples. Fig (7a) shows the monotonically distributing granular structures of less than $1\mu\text{m}$ noticed in pure PANI [37]. Micrograph of PANI in Fig (7a) shows the agglomerated globular structure [38]. The FESEM profile of Y_2O_3 in Figure (7b) shows agglomerated globular with random grain orientation. The Y_2O_3 exposed parts agglomerating due to the interaction dipole. The composites shown in Fig 7c display a morphology distinct from that of pure and bare PANI. The polymerization of aniline over Y_2O_3 particles PANI- Y_2O_3 -20 wt % is observed with globular agglomeration. Such a

morphology is very favorable to the application of gas and moisture detection and to the reading various electrical and electromagnetic properties of the PANI and its composites [39]. Fig (8d) reveals EDX energy dispersion X-ray spectrum, presenting PANI carbon, nitrogen peaks in addition yttrium oxygen, yttrium peaks of yttrium oxide in an appropriate ratio. Thus, the distinct peaks up to the formation of the composite and the elementary composition is in close competition with that of the captured composition in the course of the composite PANI- Y_2O_3 -20 wt% synthesis, the characteristic of the elementary composition entered by EDX.

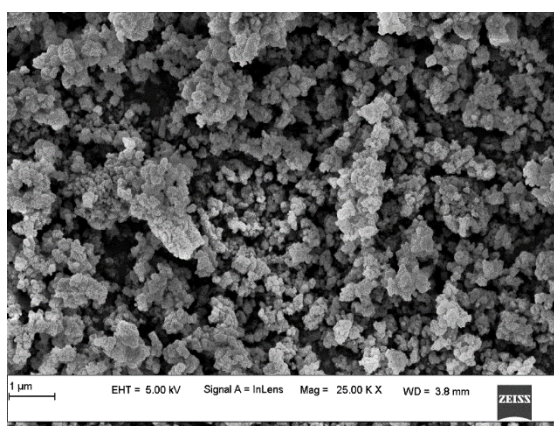




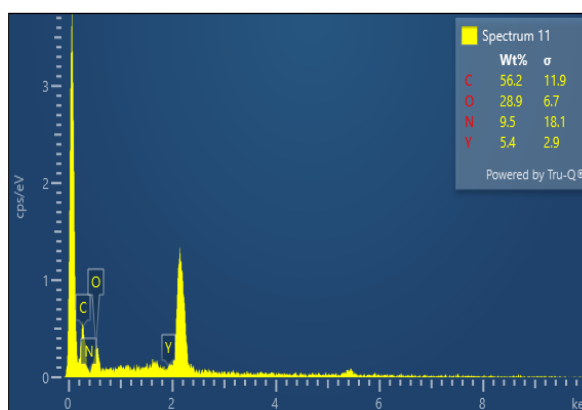
(e)



(f)



(i)



(g)

Fig 7: FESEM images

a) Pure PANI b) Pure Y_2O_3 c) PANI- Y_2O_3 -5% d) PANI- Y_2O_3 -10% e) PANI- Y_2O_3 -15% f) PANI- Y_2O_3 -20% g) PANI- Y_2O_3 -25% composites and i) EDX image of PANI- Y_2O_3 -20% composite

4. Conclusions:

Polyaniline and PANI- Y_2O_3 composites have been extensively studied due to their various properties, additionally with their environmental stability and shown much favourable to humidity and gas sensing elements. Yttrium oxide doped PANI was effectively synthesized using the in situ polymerization method. It is clear from XRD data that the structure of the composite with a semi-crystalline pattern was observed as the weight percentage of Y_2O_3 composites increases. TG-DSC analysis recommends a certain thermal stability

of composites. It is observed that the thermal degradation temperature of PANI- Y_2O_3 composites is heightened with the addition of Y_2O_3 in the composites due to the strong interconnection between Y_2O_3 and the polymer chain. DSC curves show that two endothermic dips appear in PANI- Y_2O_3 composites as the increase in the endothermic dip of composites is caused by the strong intermolecular interaction of Y_2O_3 particles with PANI. This indicates that the sample was in an amorphous state with semi-crystalline. The FTIR spectra comply with the presence of Y_2O_3 in PANI, and there is a Van-der Waals' interaction between Y_2O_3

and polyaniline particles during the composite development. The FESEM and EDX images give about the elemental analysis morphology of the PANI and PANI-Y₂O₃ composites, showed the globular agglomeration with random grain orientation. This leads PANI-Y₂O₃ composites in many fields of applications as in electronics, biology and in semiconductor devices.

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