Physical Properties of Polyacrylamide/Polyvinylalcohol Silica Nanocomposites

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Abstract: In this paper, samples of Polyacrylamide (PAAm) nanosilica nanocomposites were prepared having different concentration of nanosilica (0.125, 0.25, and 0.5). Polyacrylamide (PAAm) and poly (vinyl alcohol) (PVA) were blended with different ratio (3/1, 1/1, 1/3) using solution-cast technique. The prepared films were characterized by Fourier transform infrared (FTIR), X-ray diffractions (XRD) and scanning electron microscopy (SEM). FTIR spectra showed the presence of hydrogen bonding between $-CONH_2$ groups in PAAm and $-OH$ group in PVA and confirm the hydrophilic nature of the blends. X-ray diffractions shows the presence of a strong broad peak centered at 22° (2θ) confirms the amorphous nature of silica which is supposed to be the characteristic of SiO_2. The results obtained from different experimental techniques were supported by SEM image analysis. The thermal stability of the nanocomposites enhanced by increasing the silica content in the blend. The DC electrical conductivity was studied for all prepared samples. It was found that the conductivity increase by increasing silica content as well as, increased by increasing the wt% of PVA.

Keywords: Polyacrylamide (PAAm), Polyvinylalcohol (PVA), Nanosilica SiO_2, Blend, Thermal stability TGA and Electrical conductivity.

1. INTRODUCTION

Polymer matrix reinforced by nanofiller is probably the most commonly used form of composites in structural application, such as air craft's, boats, automobiles [1]. A single material may not meet all the requirements necessary for a potential filler. Since the final properties of the composites are controlled by the properties and proportion of the component materials, the choice of the filler plays a very important role in polymer composites [2, 3].

Polyacrylamide (PAAm) nanosilica inclusions composites were prepared having different ratios of nanosilica. The silica inclusions were either modified or unmodified. The nanocomposite films were characterized by transmission electron microscope (TEM) and infrared spectroscopy (IR). The dielectric behaviour of the composites had been investigated as a function of the concentration of the nanosilica inclusions. The results show that the dielectric constant and dielectric loss of the nanocomposites are smaller than those obtained for the neat polymer. As the concentration of the surface-modified nanosilica inclusions increases, the dielectric constant decreases. This behaviour may be attributed to the successful interaction between the polymer and the surface of the nanosilica inclusions [4]. Poly (vinyl alcohol) (PVA) is a low cost hydrophilic polymer, and shows potential application as hydrogel, polyelectrolytes, optical

materials, and biomaterials [5-7]. A large number of research groups have reported on the fabrication of organic inorganic hybrid membranes containing inorganic oxides SiO_2, which has contributed to the improvement of chemical and mechanical properties. In most cases, inorganic oxides were added into the polymer matrix using a sol-gel process from inorganic precursors [8-11]. As we all know that the homogeneous distribution of inorganic oxides in the resulting organic-inorganic hybrid membranes can increase the mechanical properties, however the interaction of polymer and inorganic nanoparticles also plays a great role in the increase of the properties of the hybrid materials [12]. The crystallinity decreased with increased silica content and changes the morphology and structure due to the formation of cross-linking [13].

Experimental investigations were carried out in polymer blends have gained significant importance, studies on several polymer blends [14], and although the formation of a single phase, homogeneous, compatible blends is thermodynamically unfavourable. Several experimental techniques have been employed to study the polymer blends such as dielectric relaxation [15, 16], nuclear magnetic resonance [17, 18] and electron spin resonance [19]. Thermally stimulated discharge current (TSDC) studies in polyacrylamide(PAAm) and the electrical conduction phenomena and TSDC studies in polyvinyl alcohol(PVA) in the low temperature have been reported earlier [20-24]. The thermally stimulated luminescence (TSL) in PAAm-PVA blends have been made and results are correlated with the results

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obtained from differential scanning calorimetry (DSC) and Optical microphotography [25]. Thermal behavior of poly (vinyl alcohol)-graft-polyacrylamide copolymers (PVA-g-PAAm), so-called intramolecular polymer-polymer complexes was studied. Complete compatibility of PVA and PAAm through hydrogen bonding is realized in the range of the graft lengths not exceeding some critical value. The content of adsorbed and trapped water in graft copolymers reflects some features of PVA-g-PAAm structure depending on the graft lengths [26].

The aim of the research focuses on the preparation of PAAm in presence of different concentration of nanosilica (0.125, 0.25 and 0.5) and preparation of PAAm/PVA blend nanocomposites with different ratio (3/1,1/1,1/3).Furthermore, morphological studies of forming nanocomposites have been conducted using X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The effects of adding different ratio of PVA and concentration of SiO_2 on thermal stability and the electrical conductivity of PAAm was also studied.

2. EXPERIMENTAL

2.1. Materials

The raw materials used in our experiments are available as commercial products. Polyvinyl alcohol (PVA) (mol. wt =5417), Polyacrylamide (PAAm) in the form of powder (mol. wt = 5×10^6).Silica nanoparticle 27 nm.

2.2. Preparation of PAAm/PVA Blend

Both PAAm and PVA are water soluble resins. Then polymer blends of PAAm-PVA with different ratio are obtained by evaporation of a solution of these polymers on petri dishes, to yield stable polymer blend transparent films as shown in (Table **1**).

2.3. Preparation of PAAm/PVA/ Silica Nanoparticle (Fumed Silica SiO_2)

Different weight percent (0.125, 0.250, 0.500 wt %) of silica nanoparticle was added to PAAm and PAAm/PVA blends in sonicator at 30 min at room temperature as shown in (Table **1**).

2.4. Characterization

Fourier Transform Infrared (FTIR)

IR measurement was carried out with KBr methods PAAm/PVA blends and PAAm/PVA nanocomposite samples (2 mg), which was dried overnight at 60°C under reduced pressure, were mechanically well-blended with 100 mg of KBr. The thickness of the KBr disk was 0.5 mm. The KBr disk of the mixed powder was desiccated for 24 h at 110°C under reduced

Table 1:   Composition of PAAm/PVA Blends and their Silica Nanocomposites

Sample Code	PAAm	PVA	Nanosilica Conc.
M0	80gm	-	0
M1	80gm	-	0.125
M2	80gm	-	0.250
M3	80gm	-	0.500
MA0	60gm	20gm	0
MA1	60gm	20gm	0.125
MA2	60gm	20gm	0.250
MA3	60gm	20gm	0.500
MB0	40gm	40gm	0
MB1	40gm	40gm	0.125
MB2	40gm	40gm	0.250
MB3	40gm	40gm	0.50
MC0	20gm	60gm	0
MC1	20gm	60gm	0.125
MC2	20gm	60gm	0.250
MC3	20gm	60gm	0.50

pressure and then its IR spectrum was recorded with a Shimadzu FTIR-4200 spectrometer using a disk of 100 mg KBr as a reference. The maximum intensity of the IR absorption band was determined by the baseline method.

X-Ray Diffraction (XRD)

X-ray diffraction (XRD) patterns of the sheet samples were recorded on X-ray diffractometer (D/Max2500VB2+/Pc, Rigaku Company, Tokyo, Japan) with area detector operating at a voltage of 40 kV and a current of 50 mA using Cu Kα radiation (λ = 0.154 nm). The scanning rate was 1°/min and the scanning scope of 2θ was from 5° to 50° at room temperature.

Scanning Electron Microscope (SEM)

The micro structure of samples was examined from micrographs taken with (SEM, TOPCON ABT150S, Japan). Samples were mounted on metal grids and coated with gold under vacuum before observation.

Thermogravimetric Analysis (TGA)

TGA measurements were carried out on a Netzsch DSC 204 (Germany). Traces of each sample were obtained from the second heating run at a constant rate of $10^{o}C$/min, after the first run of heating up to 700°C and cooling naturally at room temperature under nitrogen atmosphere.

The Electrical Conductivity Measurements

The conductivity measurements were carried out by four-probe technique recorded by Keithley electrometer type 6517 A. The pellet used in this measurement is placed between two copper electrodes; which were connected to the two terminal of Keithley electrometer. The samples were pressed into pellets of 1.33 cm diameter and 1 mm thickness in a hydraulic press

(Kimaya Engineers; India; Model WT-324) at five metric ton pressure.

3. RESULTS AND DISCUSSION

3.1. Fourier Transform Infrared (FTIR)

The FTIR spectra of all samples almost was similar so the sample MB3 was chosen. The band at 3435 cm^{-1} corresponded to the O-H stretching vibrations and N-H stretching primary amine (Figure **1**). The peak at 1663cm^{-1} due to C=O of PAAmAmorphous silica exhibits a relatively strong peak around the peaks at 1112 and 804 cm^{-1} due to the O-Si-O asymmetric and symmetric stretching modes, respectively. Addition of PVA into PAAm polymer matrix, bands of PAAm at 3124 cm^{-1} and 3248 cm^{-1} which are symmetric and asymmetric stretching vibration of –NH$_2$ group involved in inter molecular interaction of hydrogen bond coupled with –OH group of PVA at 3145 cm^{-1} and 3301 cm^{-1} [27, 28].

3.2. X-Ray Diffraction

For clarification, the XRD of MB3 sample was examined and shown in (Figure **2**) to confirm the presence of nanosilica. A strong broad peak centered at (2θ) 22° confirms the amorphous nature of silica which is supposed to be the characteristic of SiO$_2$. Due to thermochemical process, the crystalline particles transforms into amorphous silica which is indicated by broad peak [27, 28].

3.3. Scanning Electron Microscope (SEM)

In general, the nanosilica particle is shown in Figure **3a**. The morphology of nanosilica is observed as spherical in shape [29] and it exhibits the agglomerated particle with uniform size in the range of 90 nm. The
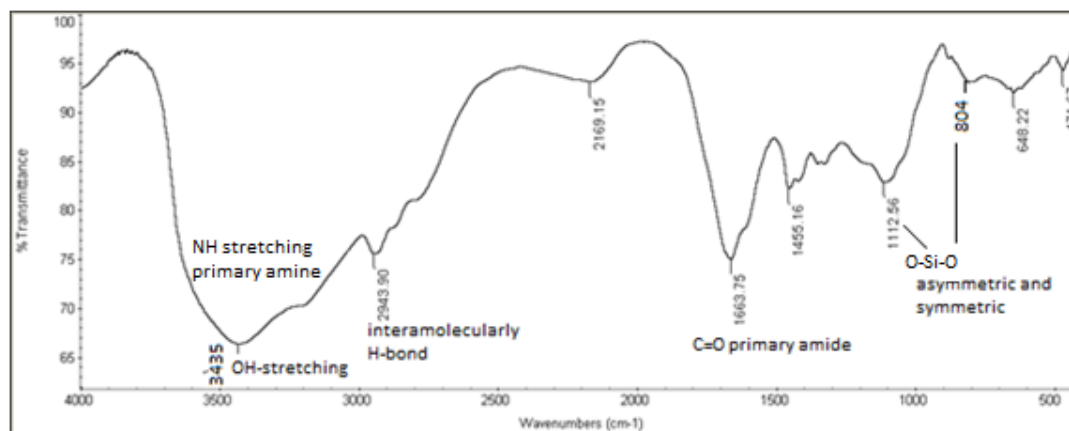

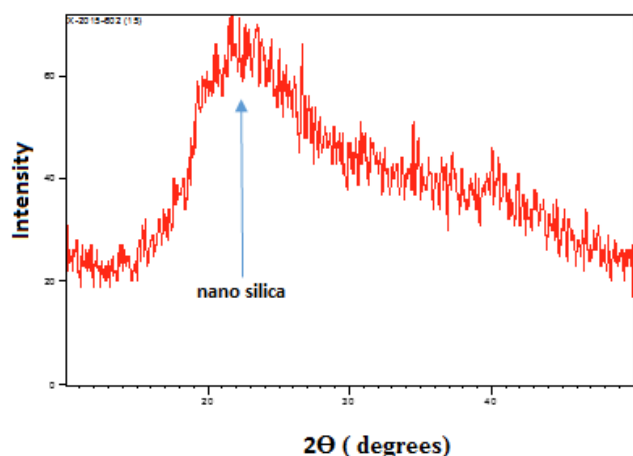**Figure 1:** FTIR spectrum of MB3.

Figure 2: XRD spectrum of MB3 sample.

presence of the nano-sized spherical particles leads to a highly porous structure. The scanning electron microscopy (SEM) shows microstructure morphologies of polyacrylamide PAAm and polyvinylalcohol PVA as shown in (Figure **3b, c**). SEM images for PAAm show smooth and homogeneous surface with some strips but PVA show as sponge structure. Formation of homogeneous blends was mostly caused by the interaction of hydrogen bonds between the functional groups of blend components [30]. As shown in (Figure **3d-f**), the blend surfaces have heterogeneous mesh type morphology. This is possibly due to PVA, linear polymer, cross-linked with PAAm may form clusters/domains of chains bonding via hydrogen bond between $-CONH_2$ groups in PAAm and $-OH$ group in PVA. At low PVA content, cross-linking density is low. So the network chains have good movement and arrange themselves to make mesh type of domain

network. As PVA content increases, the mesh/domain size keeps shrinking. From SEM micrograph, the interaction between PAAm and PVA is much greater than others and the surface of 1/1 blend ratio is rougher than the other blends. This also indicates the enhancement in thermal and electrical properties [31]. In addition the presence of nanosilica increase the homogeneity mesh type morphology of the blend surface. Formation of homogeneous blends was mostly caused by the interaction of hydrogen bonds between the functional groups of blend components.

3.4. Thermal Stability and Degradation

The thermal degradation behaviour of pure PAAm and their blend samples of PAAm/PVA in presence of different concentration of nanosilica were examined by TGA. The addition of nanoparticles can be used to manipulate the optical, thermal, magnetic, and electric properties of polymers [32, 33]. Compared with pure polymers, the polymer composite materials containing silica nanoparticles may exhibit more satisfactory thermal stability, toughness and strength owing to the effect of physical adsorption such as hydrogen bond formation [34, 35].

The effect of different concentration of nanosilica on the thermal stability of PAAm was shown in (Figure **4**), the first stage decomposition was due to the loss of absorbed water molecules until $280^{O}C$ and the next stage decomposition included the major weight losses. It was observed that with increasing the concentration of nanosilica in PAAm increase T_{10}, T_{50}, T_{max} and also the char residue % respect in compared with pure
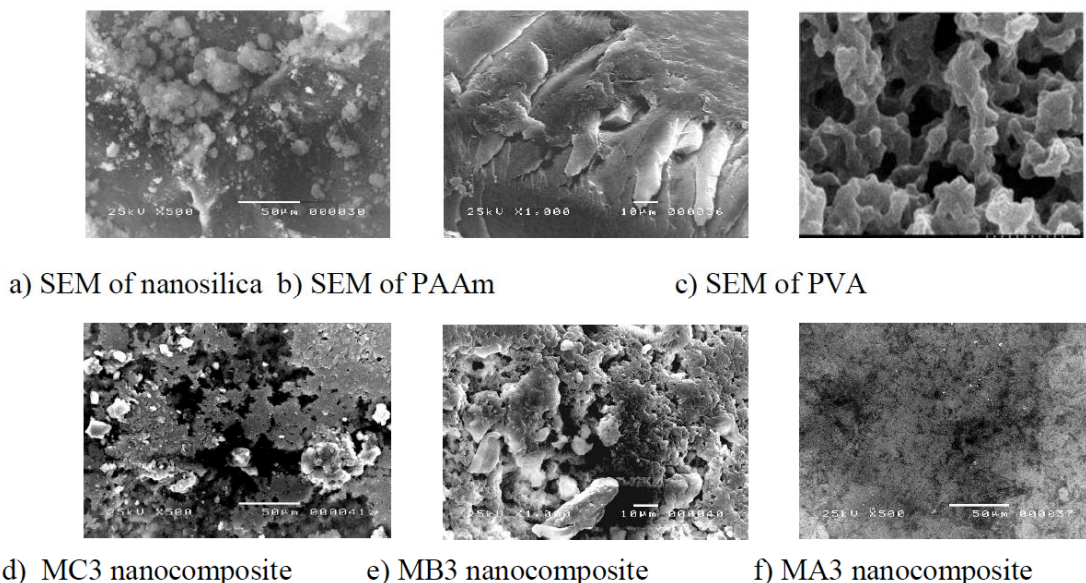

a) SEM of nanosilica   b) SEM of PAAm   c) SEM of PVA

d) MC3 nanocomposite   e) MB3 nanocomposite   f) MA3 nanocomposite

Figure 3: Scanning electron micrographs.

PAAm as shown in (Table **2**). The main reason for these results is that silica nanoparticles can interact with PAAm molecules by the formation of hydrogen bonds between the silanol functions of the silica and the amide groups in the PAAm molecular chain. These interactions can reinforce the polymer structure and increase the thermal stability. With increasing concentration of silica, the fraction of amide groups interacting with the nanoparticles becomes stronger, consequently, the movement of polymer chains is limited, and the thermal stability increases [36].
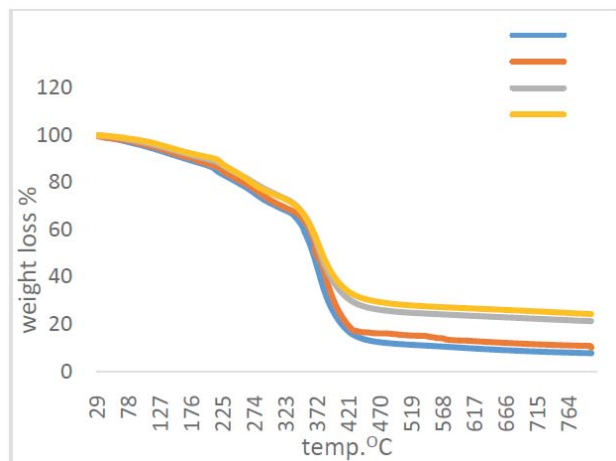

Figure 4: TGA of PAAm in presence of different concentration of nanosilica.

Depending on this results we selected the concentration 0.5% of nanosilica to study the effect of different ratio of PAAm/PVA blend as shown in (Figure **5**). The addition of PVA with different ratio to PAAm decreased the thermal stability of PAAm. This could correspond to structural decomposition of polymer blends and higher weight loss in this region which indicated the existence of chemical degradation. Relevant data are shown in (Table **3**). This higher thermal stability was observed for 3:1 blend sample by TGA was due to the intermolecular cross-linking reaction giving highly compatible impact blend system. In contrast in ref. [31, 37, 38] as the concentration of PVA increase the thermal stability decreased in the blend. The data obtained by TGA indicated the

possibility of a strong hydrogen bonding between PAAm and PVA due to $-CONH_2$ groups in PAAm and $-OH$ group in PVA, which was also confirmed by our FTIR study.
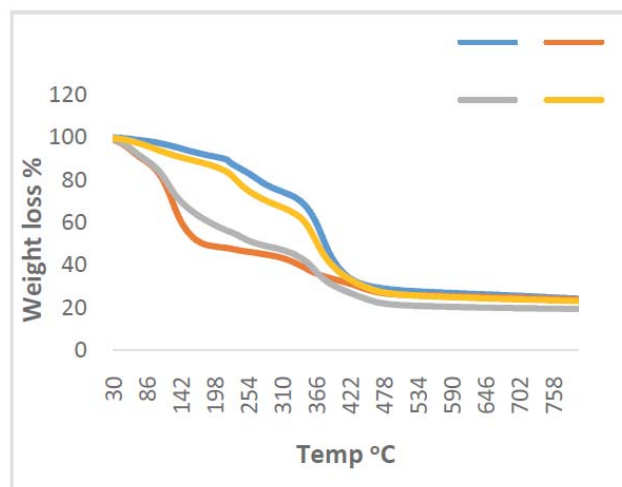

Figure 5: Effect of different ratio of PAAm/ PVA blend at constant concentration of nanosilica (0.5 %).

By studying the PAAm/PVA with ratio 3:1 with different concentration of nanosilica, it was observed by increasing the concentration of nanosilica the thermal stability was increased as shown in (Figure **6**). The main reason for these results is that silica nanoparticles can interact with this blend molecules by the formation of hydrogen bonds between the silanol functions of the silica and the amide groups in the PAAm molecular chain in the blend [36] as shown in (Table **4**).

From TGA, we may conclude that the thermal stability regions of the PAAm/nanosilica samples are higher than the PAAm pure and its stability is enhanced by increasing the concentration of nanosilica in PAAm polymer matrix and it becomes more stable at 0.5%. of nanosilica.

3.5. The Electrical Conductivity of PAAm/ PVA/SiO$_2$ Nanocomposites

(Figure **7**) shows the temperature dependence of the dc conductivity of PAAm/SiO$_2$ nanocomposites

Table 2:   Effect of Different Concentration of Nanosilica on PAAm

Sample	T_{10}	T_{50}	T_{max}	Char Residue %
M0	167	366	375	7.61
M1	182	370	373	10
M2	199	377	373	21.11
M3	213	380	376	24.16

Table 3: **Effect of Different Ratio of PAAm/ PVA Blend at Constant Concentration of Nanosilica (0.5 %)**

Sample	T_{10}	T_{50}	T_{max}	Char Residue %
M3	202	381	376.37	24.16
MA3	140	358	365.56	23.13
MB3	70	258	364.21	19.39
MC3	68	169	438.52	18.74

samples. The pattern indicates an increase in conductivity with rise in temperature. This type of temperature dependence indicates that the electrical conduction in the samples is a thermally activated transport process governed by Arrhenius relation $\sigma_{dc} = \sigma_0 \exp(-E_a/kT)$ where σ_0 is the dc conductivity pre-exponential factor and E_a, the activation energy [39].
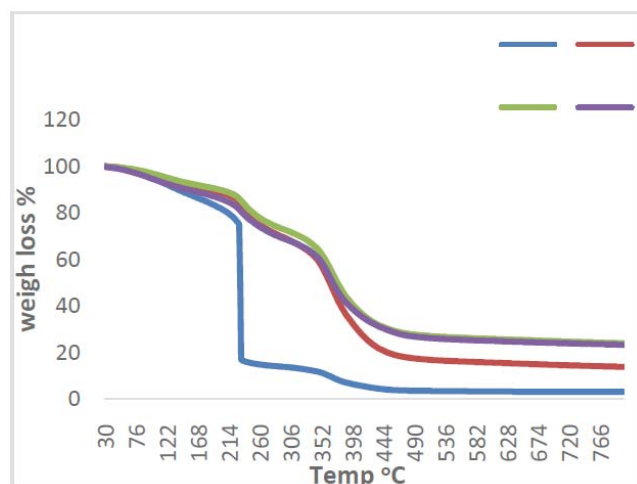

Figure 6: Effect of different concentration of nanosilica of PAAm /PVA blend at constant Ratio (3:1).

Figure **8** shows the variation of the d.c. conductivity values at two different temperatures (25, 80 °C) for the PAAm/silica nanocomposites versus the contents of silica (wt%). The Figure elucidates that the of the nanocomposites is higher than that of the neat PAAm, also it increases with increasing the silica content in the nanocomposites till 0.25 % of the silica content then decrease at high concentration (0.5 %). The silica surface contains hydroxyl groups, which makes its

surface polar. According to Wolff and Wang [40], the surface energies of silica are characterized by a lower dispersive component and a higher specific component. The lower dispersive components cause weak polymer–filler interactions while higher specific component leads to strong interactions among the silica particles. Moreover, at the same time, the polar surface of silica enhances filler–filler interactions also resulting in strong intermolecular hydrogen bonds between hydroxyl groups of silica [41, 42]. Due to such strong interactions, it can aggregate tightly resulting in its poor dispersion in the PAAm nanocomposites which explains the decrease in the with the increase of silica filler contents at higher concentration of the silica [43, 44].

The effect of the PVA weight fraction on electrical conductivity of PAAm/PVA/SiO$_2$ blend nanocomposites at two temperatures (25, 80 °C) is shown in Figure **9**. The figure shows that the electrical conductivity increases with increasing the weight fraction of PVA. It is known that under the applied electrical field the PVA, similarly as many other dielectric polymers, is polarized [45]. The polarization is due to the space charge formed by the carriers which were injected to the polymer from the electrodes and trapped in the bulk, dipolar orientation, migrational polarization, etc.

4. CONCLUSION

Polyacrylamide (PAAm) nanosilica nanocomposites were prepared having different ratios of nanosilica (0.125%, 0.25%, and 0.5%). Compatible PAAm/PVA blends were prepared successfully using solution-cast

Table 4: **Effect of Different Concentration of Nanosilica of PAAm /PVA Blend at Constant Ratio (3:1)**

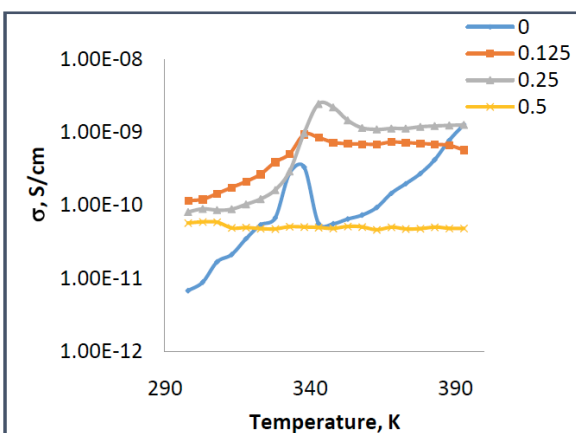| Sample	T_{10}	T_{50}	T_{max}	Char Residue%
MA0	136	230	229	3.03
MA1	178	363	368	13.69
MA2	197	375	366.91	23.81
MA3	150	368	365.56	23.13

Figure 7: Variation of electrical conductivity of the PAAm/SiO$_2$ nanocomposites with the temperature.
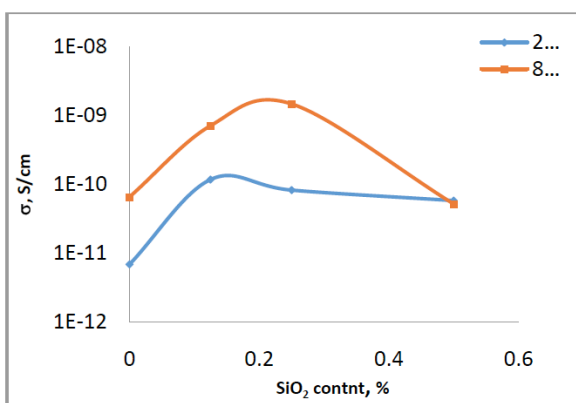

Figure 8: Effect of silica content on the Electrical conductivity of the PAAm /SiO$_2$ nanocomposites at two different temperatures.
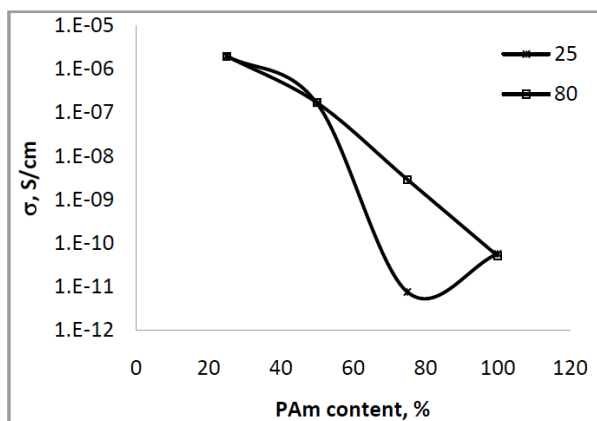

Figure 9: The effect of PVA content on the electrical conductivity of the PAAm/PVA /SiO2 nanocomposites at 0.5 silica content.

technique. FTIR analysis confirmed the conclusion about the specific hydrogen bonding interaction between –CONH$_2$ groups in PAAm and –OH group in PVA.X-ray diffractions shows the presence of a strong

broad peak centered at 22º (2θ) confirms the amorphous nature of silica which is supposed to be the characteristic of SiO$_2$.Due to this hydrogen bonding the thermal stability and electrical conductivity were improved. It was found as the concentration of nanosilica (0.5%) increase the conductivity increases and the electrical conductivity increases with increasing the weight fraction of PVA.

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