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Research Article

Study Some Properties of Hybrids Silica /PVA using Sol-Gel Method

Harith Ibrahim* and Seenaa Ibrahim*

Department of physics, college of science, university of Baghdad

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Abstract: Aim of the research study of some physical properties of the hybrid films prepared with different percentages of TEOS/PVA (20% TEOS+80% PVA, 50% TEOS +50% PVA, and 80% TEOS+20% PVA). The PVA80 (20% TEOS+80% PVA) It can be seen from these there is no variation in the transparency curve as they were in pure PVA. This means that silica has no effect on UV-Vis transparency wavelength when incorporated into the PVA. The Differential scanning calorimetric (DSC) was found that increases thermal stability of the hybrid films, an increase of ratio TEOS because of the thermal stability of silica. The last test moisture uptake, the moisture up take polyvinyl alcohol pure is more valuable than the hybrid film because the polymer hydrophilic.

Key words: TEOS, PVA, SOL-GEL, hybrids films

1. INTRODUCTION

Polymer–silica hybrids with enhanced thermal and mechanical properties (because of the silica component), better flexibility (due to the polymer content), Organic–inorganic hybrids are important in a variety of fields as they combine the desirable properties of the inorganic phase (thermal stability, rigidity) with that of the organic phase (flexibility, processability, ductility). In recent years¹. Sol-gel method has been widely employed in recent times for the synthesis of hybrid organic–inorganic composite materials². The versatility of sol-gel chemistry in polymers affords a measure of control over the nature of organic–inorganic interface and a convenient method for the introduction of newer properties such as catalysis³, biomedical⁴ and nonlinear optical properties, into the resulting material.

One important area of recent interest in the field of polymer-inorganic composites has been the synthesis of materials having hydrogel behavior². Sol-gel synthesized silica materials have a long history and are extensively reviewed⁵. There has been successful attempts to tailor the properties of the synthesized material by varying the precursors, synthesis conditions and process parameters like pH, temperature, pressure and additives/templates⁶. The overall process involves hydrolysis and polycondensation reactions of the organometallic (silicon alkoxides) or inorganic (silicic acid) compounds. Polymerization followed by condensation of siloxane bonds results in the formation of a gel which is described as a continuous solid frame work embedded in a large volume of fraction-liquid phase⁵. Polyvinyl alcohol has excellent film forming, emulsifying and adhesive properties. It is also resistant to oil, grease and solvents. It has high tensile strength and flexibility, as well as high oxygen and aroma barrier properties and PVA has a melting point of 230 °C⁷.

2. EXPERIMENTAL

2.1. Materials: All chemical materials used were of the highest purity available

1. Tetraethoxysilane (TEOS) ,Sigma-Aldrich (Germany) , the Molecular Formula $\text{Si}(\text{OC}_2\text{H}_5)_4$, Molecular Weight (g/mol) 208.3 , density =0.933, Purity >98%, State of Raw Material liquid.
2. Deionized Water (H_2O) ,University of Baghdad/ College of Science/Laboratory of service , Molecular Weight (g/mol) = 18,density = 1 ,high degree of purity/empty of additionalions ,Liquid
3. Hydrochloric Acid (HCl) ,BDH , Molecular Weight (g/mol) =36.46, density= 1.19 ,purity= 37% , Liquid.
4. Ethanol (EtOH) ,GCC/Gainland chemical company , $\text{C}_2\text{H}_5\text{OH}$ Molecular Weight (g/mol) =46.07 ,density= 0.785, purity= 99.9% Liquid
5. Polyvinylalcohol (PVA), china, $[\text{CH}_2\text{CH}(\text{OH})]_n$, Molecular Weight= 16000,It is white (colourless), State of Raw Material solid.

2-2Synthesis of pure PVA film : PVA polymer is dissolve the water , the weight of PVA (1 gm) were dissolved in (10 ml) of H_2O to give solutions of 10% (wt/vol)The solutions were stirrer for 30 minute or more at 50 °C to achieve an almost homogenous content. The samples were dried in free air for 24 hour at room temperature.

2-3Synthesis of the TEOS/PVA film: Tetraethoxysilane (TEOS) was used as the silica precursor, PVA form as the organic precursor, water(H_2O) as the solvent , hydrochloric acid (HCl) ,as the catalyst ,Three ratio of TEOS /PVA were prepared as TEOS/PVA=20/80, 50/50 and 80/20 (v/v).

PVA was dissolved in H_2O (concentration of 10 wt%). TEOS + HCl (0.1M) solution was added drop by drop to the PVA solution under continuous stirring to give three different organic/inorganic ratios (20/80, 50/50 and 80/20, v/v). The solution was stirred for 1 h at room temperature and later transferred to closed glass Petri dishes. The dishes were opened to allow the solvent to slowly evaporate at room temperature.

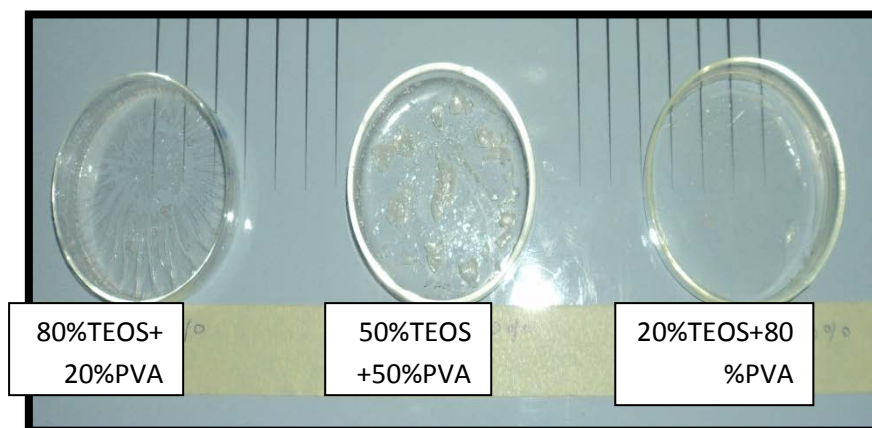


Figure 1: Samples of TEOS/PVA film

2.4. Characterization Techniques

2.4.1. UV-Visible Spectrophotometer: By using UV spectrophotometer (UV-1800/SHIMADZU) was measured transmission spectra of the hybrids samples.

2.4.2. Differential Scanning Calorimetric (DSC): Differential Scanning Calorimetric (DSC), is the heat rate $10^{\circ}\text{C}/\text{min}$.

2.4.3. Moisture Uptake: All samples were cut the $30\text{mm}\times 30\text{mm}$. The water uptake of films was calculated using the following equation⁸.

$$\text{Moisture uptake \%} = (\text{wet weight} - \text{dry weight} / \text{dry weight}) * 100\%$$

4. RESULTS AND DISSECTION

4.1. Morphology for samples: Figure (2a) shows the Morphology for prepared films, the ratio 20%TEOS+80%PVA the best homogenous compared with other ratios. Figure (2b) shows the morphology for the ratios 50%TEOS+50%PVA, and 80%TEOS+20%PVA, the films were the internal stress duo to the evaporation process and shrinkage.

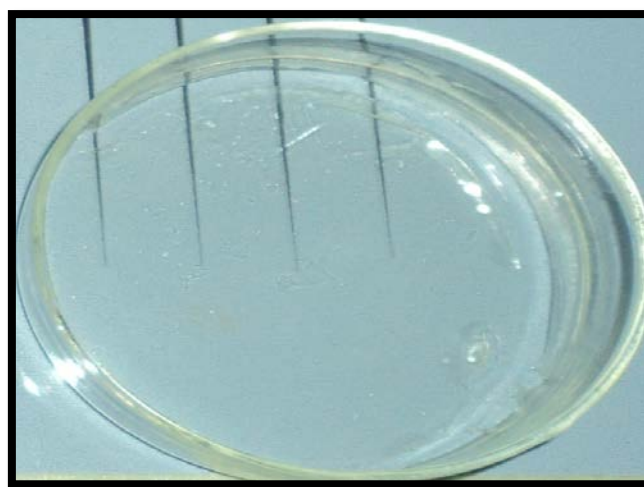
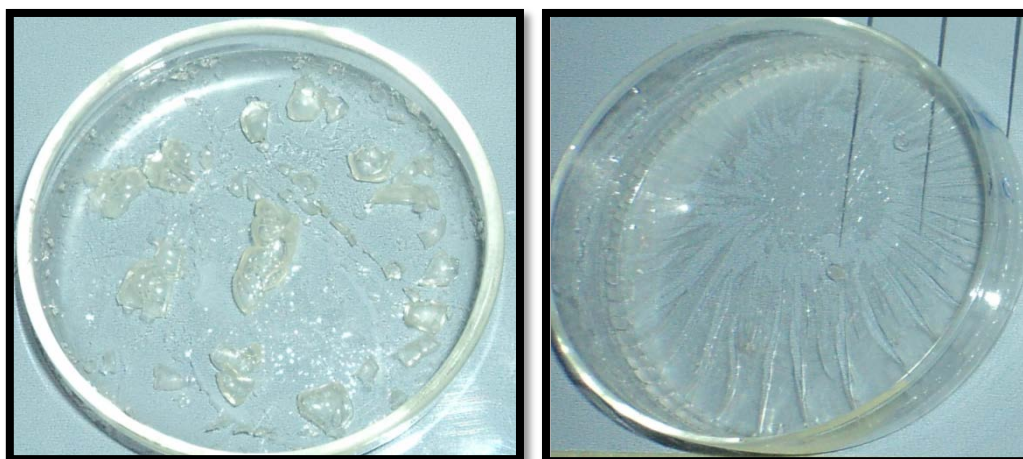


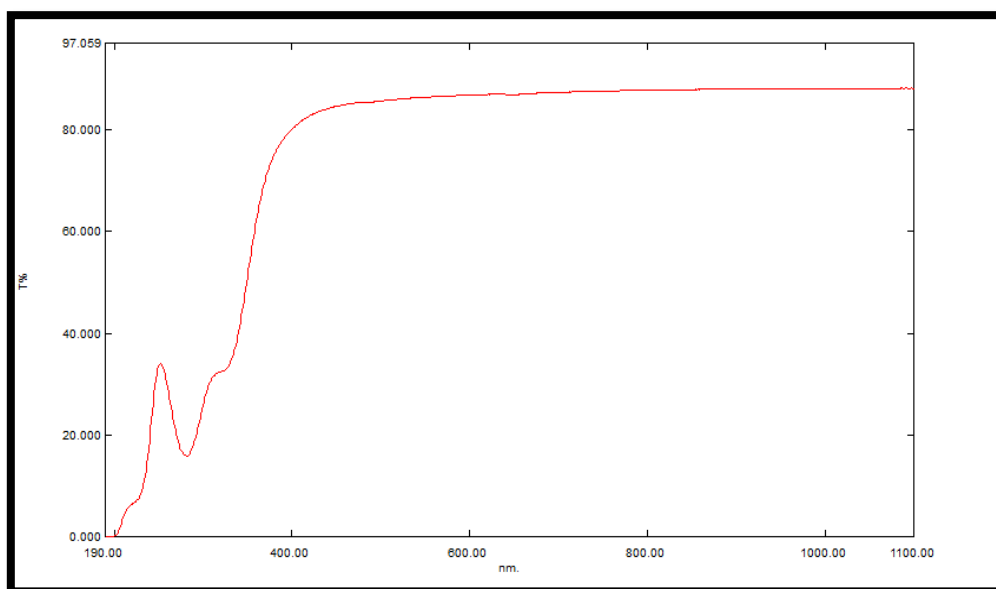
Figure 2: Morphology for film 20%TEOS +80%PVA



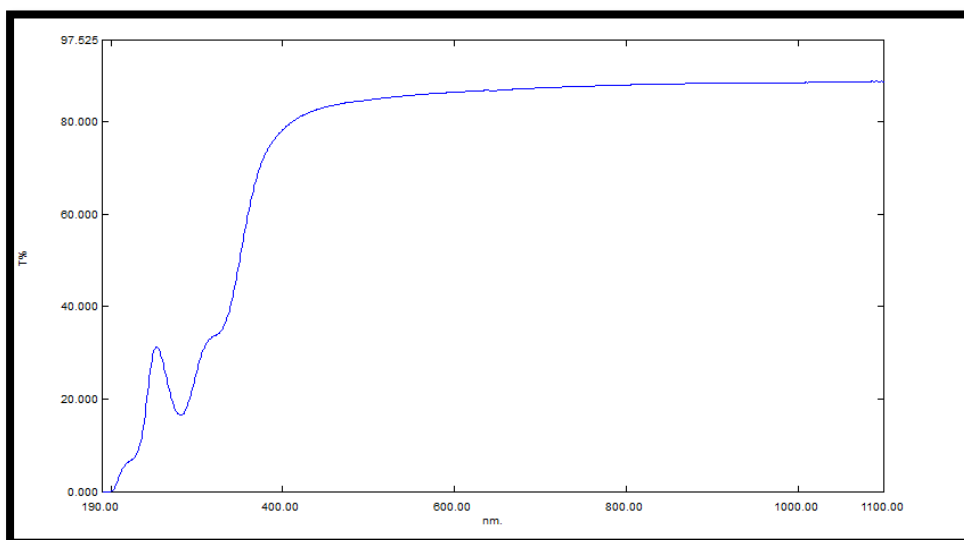
(a) 50%TEOS+50%PVA (b) 80%TEOS+20%PVA

Figure 3: Morphology for hybrids films

4.2.UV-Visible Spectrophotometer: For figure (4a) PVA pure film, high absorption was seen between 190-350nm .after that high transparency was seen between 400-1100 nm. According to figure (4b) the same behavior was observed for 20%TEOS+80%PVA, this means that silica has no effect on UV-Visible transmission when adding the polymer PVA.



(a) Transmittancecurveof PVA pure



(b) Transmittance curves of 20% TEOS & 80% PVA

Figure 4: transmittance curves of The PVA pure & ratio 20% TEOS+80% PVA film

4.3. Differential scanning calorimetric (DSC): Figure (5) The Glass transition temperature of PVA pure 50°C and the melting temperature 220°C which agree with Jeong H.Y. *et al.*⁹, when add the ratio of TEOS about PVA the thermal stability increase with increasing ratio of TEOS because the thermal stability of silica. Figure (6) shows the DSC curve from hybrid films different ratio of TEOS with PVA, DSC analyses showed that the process of interaction is endothermic reaction. Figure (6,a,b) has been observed that the area of the curve smaller than the area of curve figure(6,c) because for this to occur dehydration from the sample.

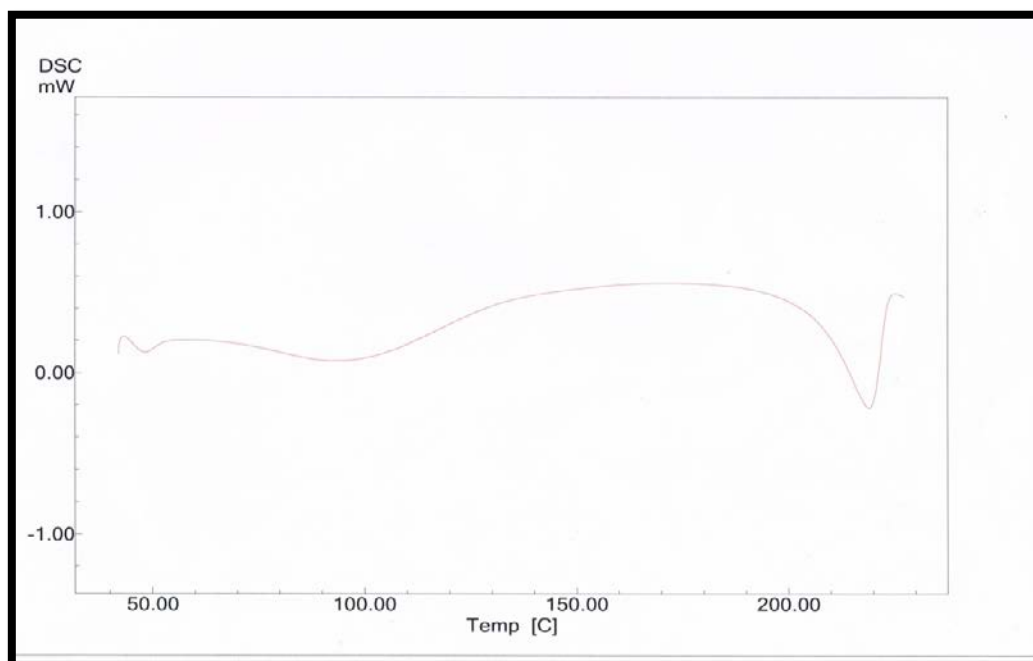
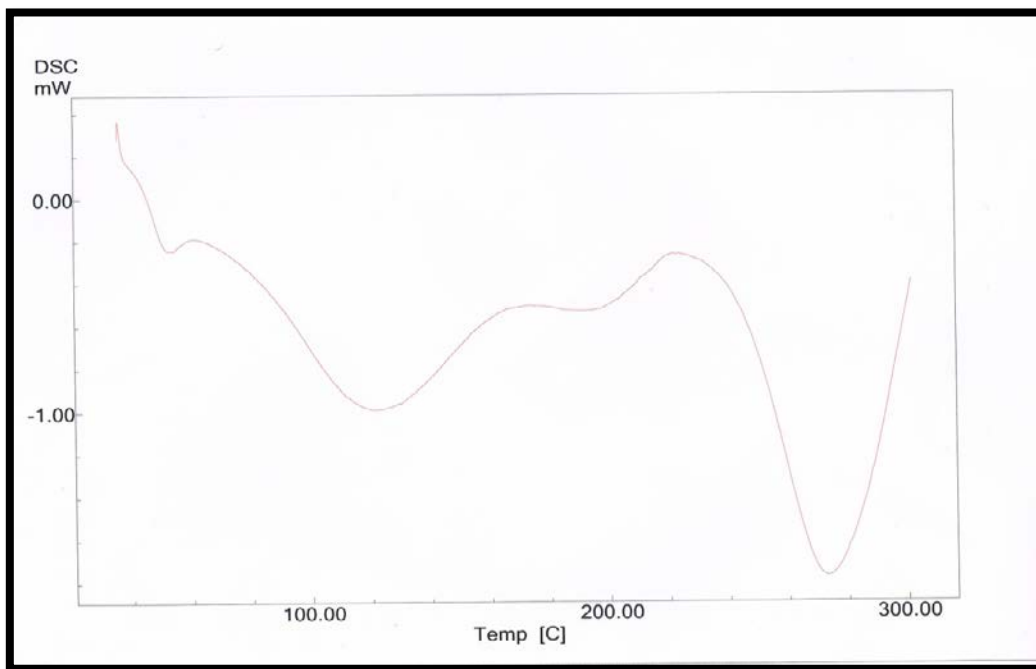
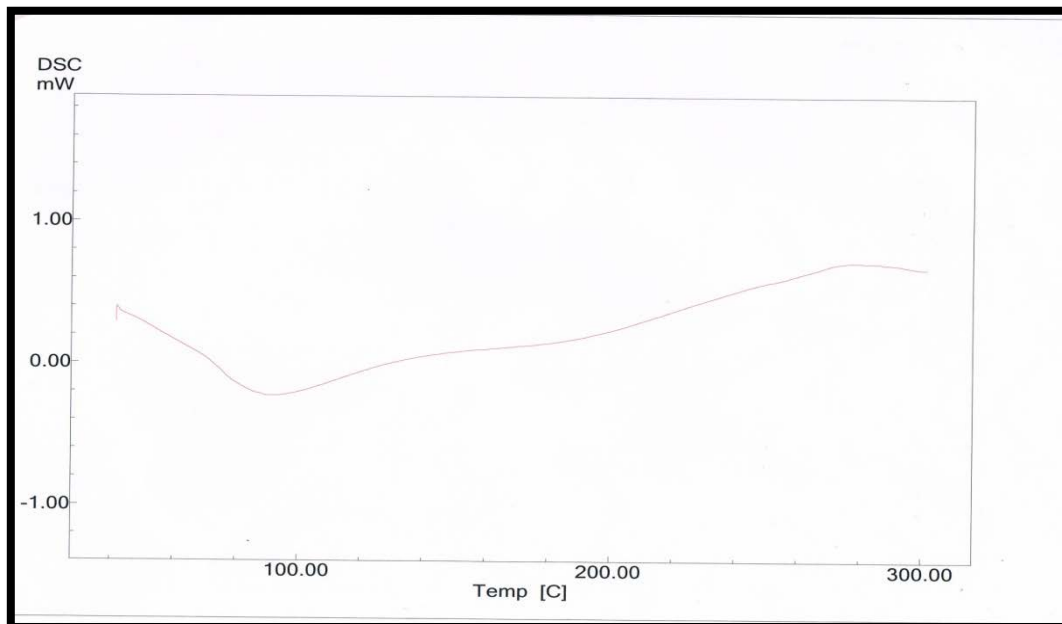


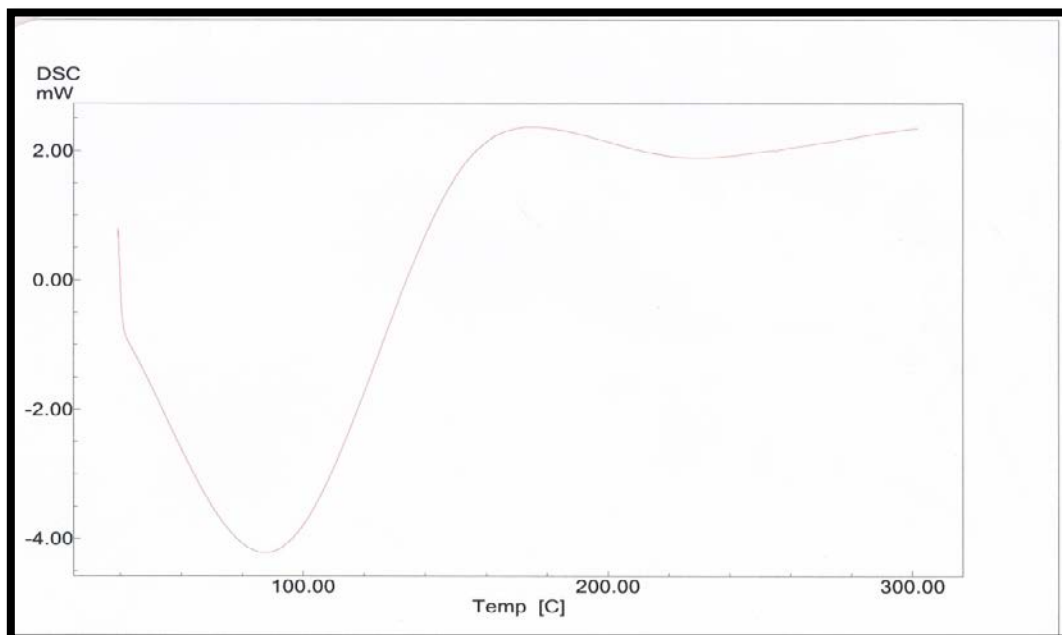
Figure 5: DSC curve of PVA pure



(a) DSC curve of 20% TEOS & 80% PVA



(b) DSC curve of 50% TEOS & 50% PVA



(C)DSC curve of 80%TEOS&20%PVA

Figure 6: DSC curve of different ratio TEOS& PVA

4.4. Moisture uptake (%): The moisture uptake (% by weight) for the hybrid film is shown in figure (7) due to the is a hydrophilic polymer¹⁰ Due hydrophobic nature of silica, the moisture uptake is decreasing with add of TEOS and moisture uptake increase with increasing the time immersed in water which agree with Muhammad H.²

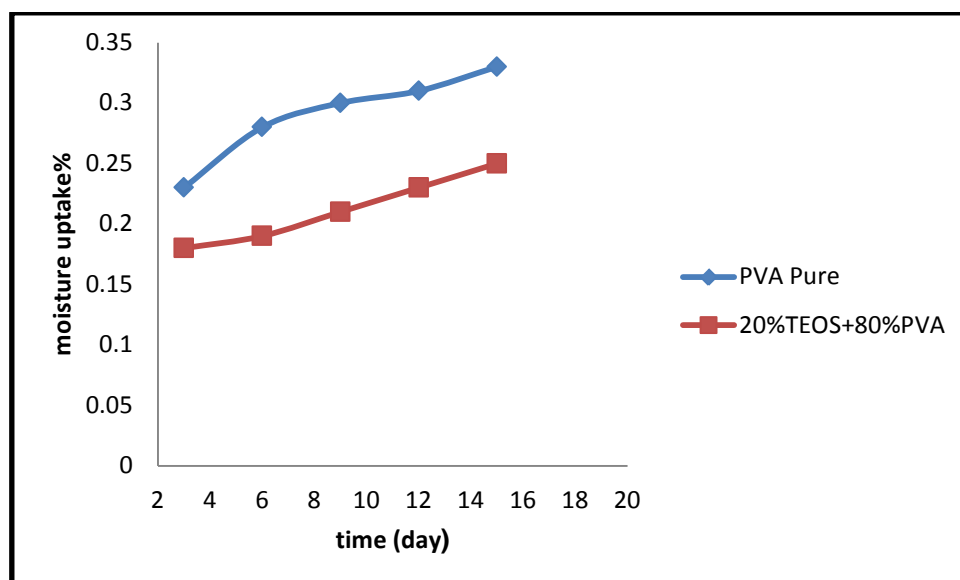


Figure 7:moisture taken up of the PVA& hybrid film (20%TEOS+80%PVA)

5. CONCLUSION

- The films are transparent in visible range i.e. 400 nm – 1100 nm while absorbance is only shown in UV- region of the spectrum. This means that there is interaction of PVA with silica only in UV-region.
- The thermal stability of the hybrid films increasing with increase the ratio of TEOS because the thermal stability of silica.
- The moisture uptake the PVA polymer more than hybrid film (20%TEOS+80%PVA), because due to is a hydrophilic polymer.

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Corresponding author:Seena Ibrahim;

Department of physics, college of science, university of Baghdad,Iraq
Harithibrahem@yahoo.com; Sinna633@gmail.com