Studying the effect of some inorganic acids on physical and mechanical properties of cross-linked (PVA) film coated polyester membrane

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I - ABSTRACT:

Cross-linked (PVA) film coated polyester membrane was prepared by using polyvinyl alcohol as a film coat and apply it on polyester fabric. The effect of some inorganic acids on the produced film coated membranes were investigated inspect of the change in its mechanical and physical properties. The produced film coated polyester membrane was found to be stable under mechanical testes when it was affected by different concentrations of some inorganic acids at different time and temperature. The physical and mechanical properties was enhanced by the coating processes and gives a good results with respect to uncoated polyester also it was enhanced by the action of inorganic acids to some extent.

Key words: polyvinyl alcohol, membrane, coated polyester membrane

II- INTRODUCTION:

Since the development of asymmetric type, membrane by (Loeb and Sourirajan, 1963)considerable progress has been made in various aspects of membrane related fields (Hassanien *et al.*, 2013;El-Aassar, 2012;Seyednejad *et al*, 2011).The interest in studies of membranes is due to the necessity of membranes with different separation properties. An asymmetrical membrane is characterized by a thin and dense top layer, commonly recognized as the skin layer, and underneath which is a porous solid matrix. It is well known that the skin layer provides major resistance to the permeation of solute through the membrane whereas the porous region functions exclusively as a mechanical support. The capability of an asymmetric to reject or admit a certain solute species is, therefore, determined by the morphology, the pore size and density of the skin layer (Wang *et al*, 2006). Accordingly, it is necessary to prepare asymmetric membranes with controllable structure of skin layer to achieve the required performance in the membrane. Poly(vinyl alcohol) (PVA) was a typical semi-crystalline and many studies have been carried out on aqueous PVA gels(Ba, 2010). Recently, the PVA membranes were coagulated with $Na_2SO_4/KOH/H_2O$ to form an asymmetric chemical stability,dmembrane(Muhammed *et al.*, 2012;Ba, 2010). Being a material with goo thermal stability, mechanical stability and high water permeability, PVA seems an attractive material for producing membranes **(**Ashrafa, *et al.*, 2013**)**. In order to control the membrane structure, low molecular weight component or the secondary polymer is frequently used as the additive in the membrane forming system **(**Cerpakovska *et al.*, 2012**)** because it offers a and effective way to develop membranes with high performances. In this present work, some of inorganic acids were used to control the membrane structure and to show the effect of these on physical and mechanical properties.

III- EXPERIMENTAL:

III-1 Materials:

Polyvinyl alcohol M.W app. (1,25,000) was supplied by Rassayan.SD-Fin-Chemical ltd.Boisar., commercial formaldehyde, Sodium sulfate supplied by ADWIC according to the method of PROLABO., sulfuric , phosphoric and nitric acid were of pure grad.

III -2 Preparation of (PVA) film coated polyester:

8% (PVA), aqueous solution cast on glass plate was dried in the air at 25^0C to obtain (10μm) thickness film. The film then thermally compressed on a partially wet polyester fabric, the film became stuck on the polyester fabric by physical bond without adhesive material due to the good adhesive power of (PVA). Then the product was cut into equal pieces.

An acidic solution of concentrated sodium sulfate containing 10 % formaldehyde and calculated amount of concentrated sulfuric acid was prepared and divided into four portions. Four pieces of film-coated polyester were soaked in the aforementioned mixtures for one hour at room temperature. Another four pieces were soaked for one hour in mixtures prepared as the above-mentioned method. The produced film coated polyester fabrics were washed several times, and soaked in distilled water to remove excessive aldehydes.

III-2.1 Testing and Analysis*:*

The produced film coated polyester fabrics were examined in the following manner:

- ***Mechanical Properties:***

• Tensile strength and elongation % at break determined by using tensile strength tester; Shimmadzu Autograph S-500.

•Abrasion resistant was determined by using rubtester; Universal Rotary Flat Abrasion Tester Type: FF -25 computext textilpari, Muszeres, Inc.

• The thickness of (PVA)film was measured by using thickness gauge micrometer; type 1264 ICV. Mahr Feinpriis, Gottingen Inc., Germany.

- ***Estimation of Moisture regain(Water content):***

Both PVA film and PVA film coated polyester membrane samples were pre-dried at 105°C for 4 h in an electric oven and then cooled in a desiccator. The samples were then put in a standard condition atmosphere for 48h at 22°C till constant weight. The moisture regain of all samples were then determined according to the following equation:

$$\text{Moisture regain } \% = \frac{W_1 - W_2}{W_2} \times 100$$

Where,

W_1 = Weight of samples after saturation in standard humidity atmosphere.

W_2= Constant weight of dry sample.

- ***Estimation of weight loss:***

Dried samples of known weight were immersed in a stopper flask containing acidic solutions of the three kind of acid (sulfuric, phosphoric and nitric acid) for different concentration namely (4 %, 6%, 8%, 10%, and 12%) using martial to liquor ratio of 1:40. The process were under taken in a shaker water bath at different temperature 20, 40, 60, and 80 °C, and a various elapsed time 30,45,and 60 minutes. Finally the samples were withdrawal, washed thoroughly and rinsed several time with distilled water and left to dry at room temperature.

The percentage of weight loss of the acid affected samples was calculated from oven - dry weights of the sample before and after the action of acid, as follows:

$$\text{weight loss} = \frac{(D - T)}{D} \times 100\ \%$$

Where:

D = Constant weight loss of the sample before acid action.

T = Constant weight loss of the sample after acid action.

IV - RESULTS AND DISCUSSION:

IV-1 - Representation of film coated polyester membrane:

When using membrane in ultra, nano, micro filtration techniques, it works under severe conditions and suffers greatly some factors; 1- It is subjected to high pressure., 2- Abrasion

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resulting from friction between membrane surface and liquid with its impurities, especially when the flow is parallel to membrane surface, and 3- Also, it is subjected to a great tension on using. To resist the above mentioned factors and to increase membrane durability, it was prepared on synthetic fabric (polyester) to support the membrane structure and improves its mechanical stability.

A schematic diagram was made to illustrate the structure of (PVA) film-coated membrane, assuming that film coated membrane can be roughly illustrated as in figure (1).

The film of (PVA) and polyester fabric are well stuck by thermally compression of partially wet polyester fabric and PVAfilm, this is due to the good adhesive power of (PVA) when moisten and dried. Now (PVA) coated film must change from water-soluble to water insoluble film, this treatment is done by aldehyde solution.

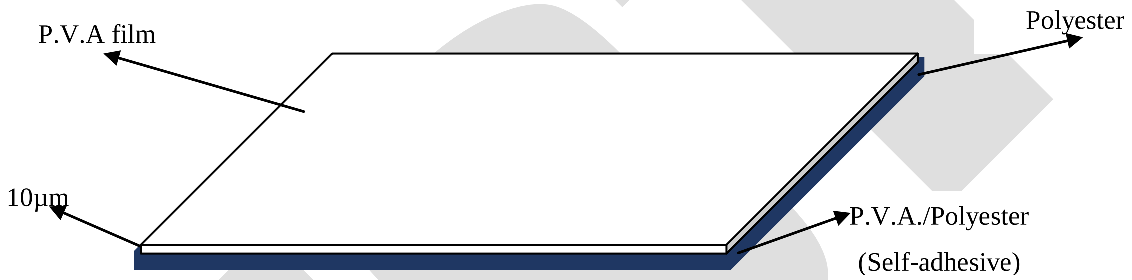
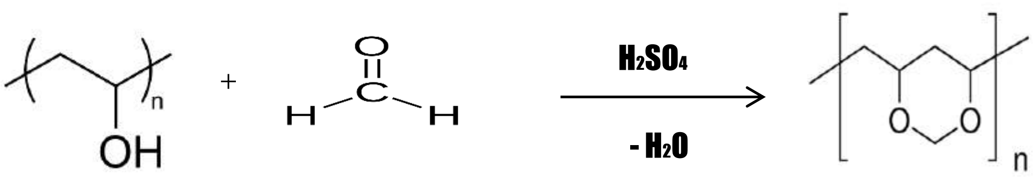


Figure (1):A schematic diagram illustrating the structure of P VA film-coated fabric membrane.

Treatment of (PVA)film coated polyester membrane with aldehyde solution in the presence of sulfuric acid asa catalyst renders the (PVA) insoluble in water due to the blocking of two adjacent(OH) groups of(PVA) and few cross -links occurs between (OH) groups of two or more neighboring chains **(**Gohil *et al*., 2006**).** This treatment of (PVA) has higher water absorption, and also found to possess excellent dimensional stability, resistance to abrasion, remarkable tensile strength, and elongation **(**Muhammad *et al*., 2015; Rajaeian, 2012; Das *et al*., 2010**).**



PVA formaldehyde polyvinylformal

IV-2-Effect of inorganic acids on the properties of film coated membrane:

To follow up changes taking place on coated polyester and uncoated one, during the action of inorganic acids by different concentration, time and temperature, a number of coated membrane were tested in accordance to physical and mechanical properties as follow :

IV- 2.1 Weight losses during the action of acids:

A pretest was carried out on freshly prepared (PVA) film and polyester fabric, by the action of acids at different concentration for different time and temperature. The results signify that the weight loss in(PVA) film does not affected by the acid within the range from (3-12%) at different time and temperature. But the action of acid on polyester fabric showed a positive and significant value of weight loss in case of sulfuric acid. While it was slightly affected by the action of Phosphoric and nitric acids.

Representative weight loss response for the film coated membrane is shown in **figures (2-10)**. It is clear that the effect of acid on film coated membrane under test expressed as weight loss increases with increasing the acid concentration at constant temperature and time. It is appear that increasing the concentration of acids (Phosphoric and Nitric acids) having weight losses less than that of Sulfuric acid at same temperature and time. The weight losses enhanced by increasing temperature and time which clearly seen from the order of curves in the **figures(2-10).**

As it is shown in **figures (11-13)** uncoated polyesters fabrics affected by 12% acidic solution at 80 °C for 60 minutes reach maximum weight loss with respect to the coated polyester fabrics in all kinds of acids. This indicate that the losses exerted in the polyester fabrics are higher than that in polyvinyl alcohol film coat, thus it can be made that the effect of acid on(PVA) film isnegligible. The data indicate that the increasing of sulfuric acid concentration showed apositive and high significant value of weight loss especially at high temperature for long time.

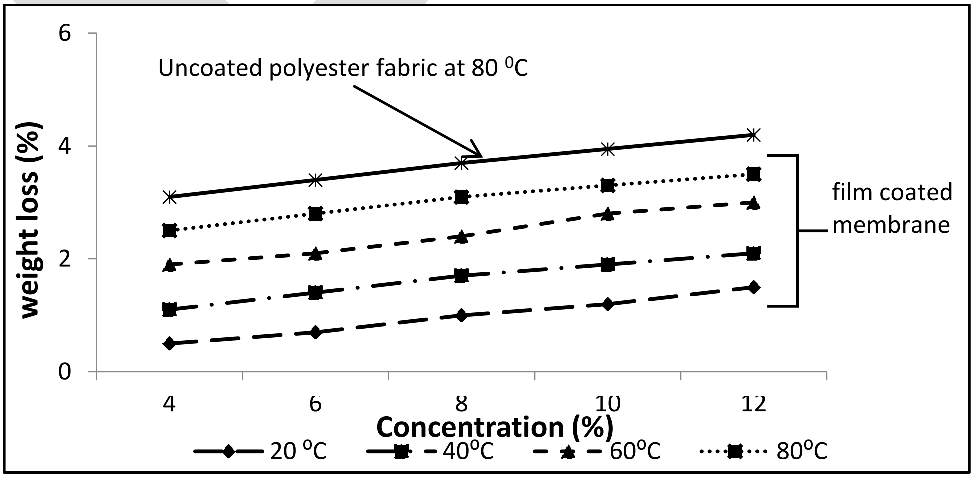


Figure (2): Effect of different concentration of nitric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 30 minutes.

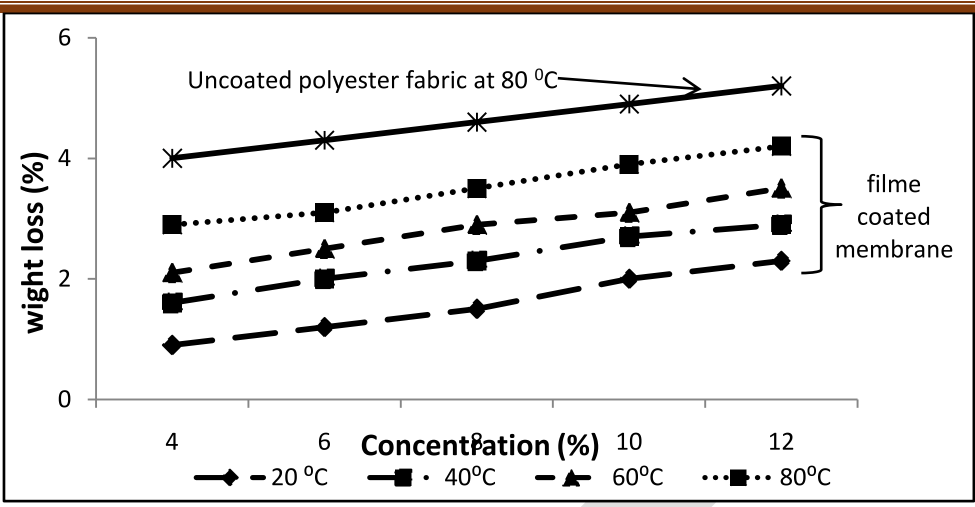


Figure (3): Effect of different concentration of nitric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 45 minutes.

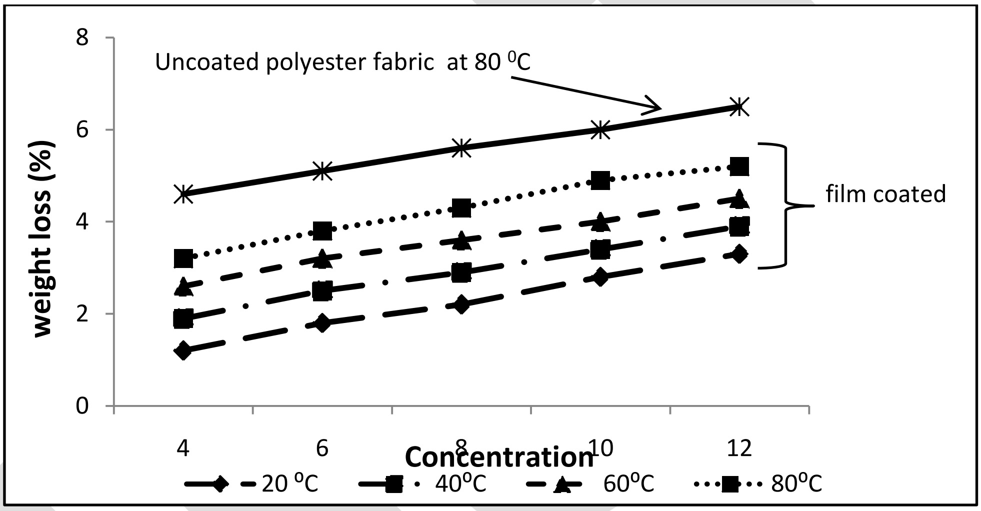


Figure (4): Effect of different concentration of nitric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 60 minutes.

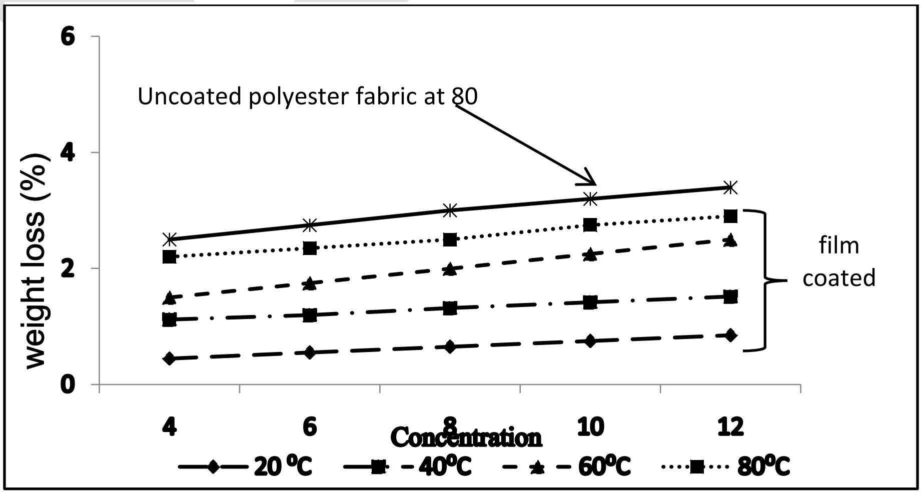


Figure (5): Effect of different concentration of phosphoric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 30 minutes.

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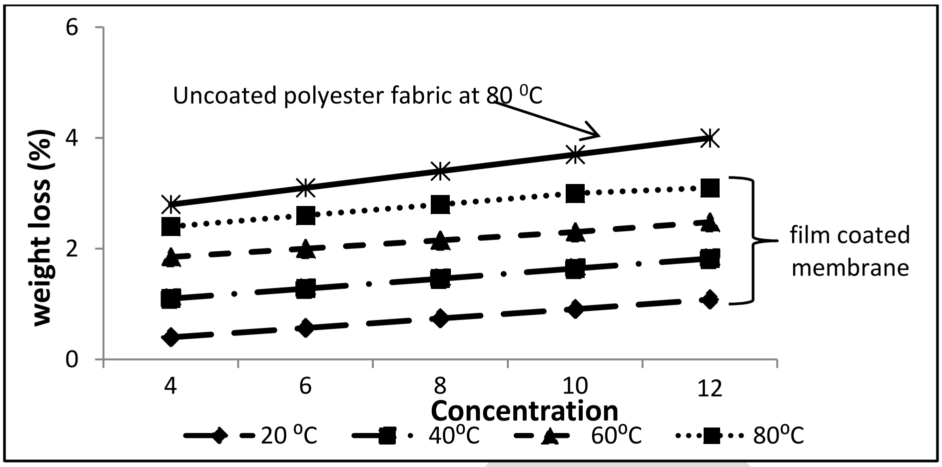
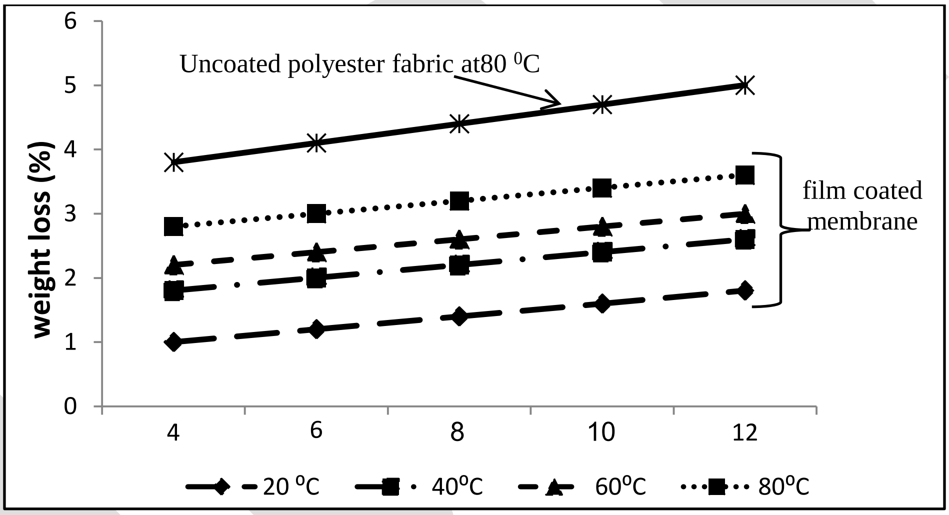


Figure (6): Effect of different concentration of phosphoric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 45 minutes.



Concentration

Figure (7): Effect of different concentration of phosphoric acid on weight loss of samples at different temperature (20ºC, 40ºC, 60ºC and 80ºC) for 60 minutes.

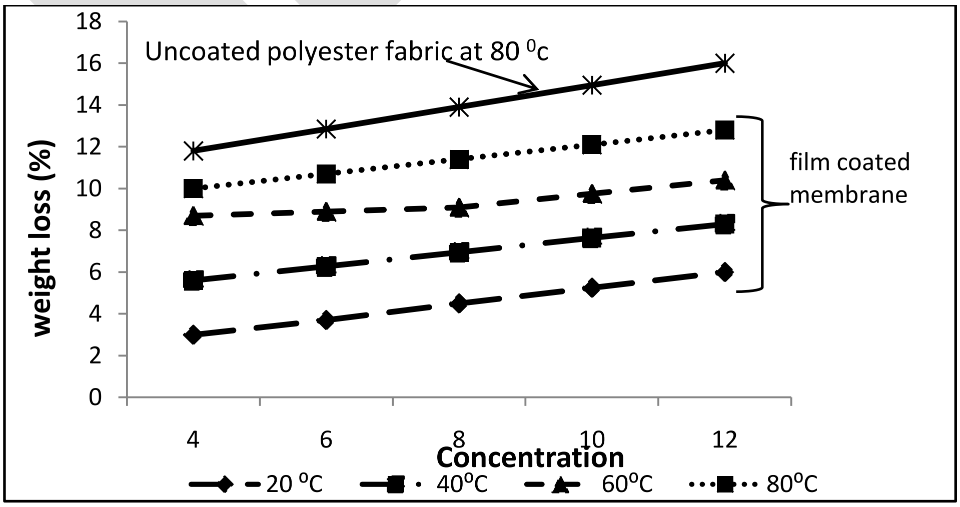


Figure (8): Effect of different concentration of sulfuric acid on weight loss of samples at different temperature (20 ºC, 40 ºC, 60 ºC and 80 ºC) for 30 minutes

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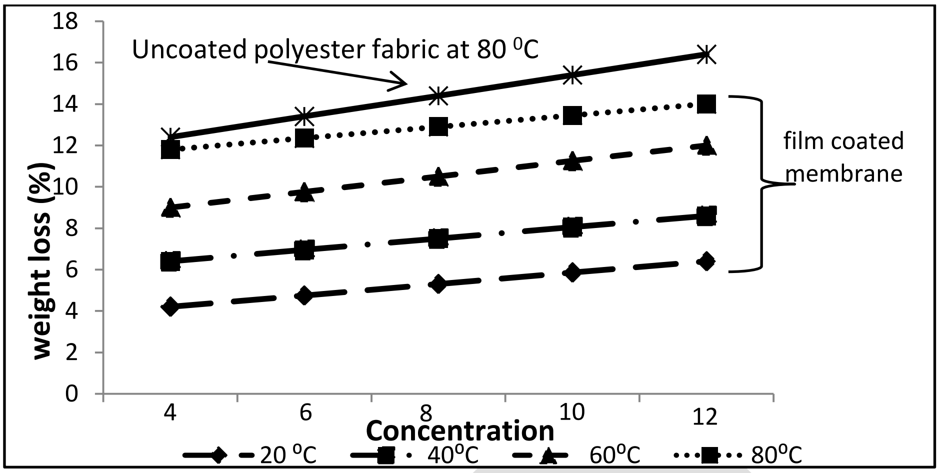


Figure (9): Effect of different concentration of sulfuric acid on weight loss of samples at different temperature (20 °C, 40 °C, 60 °C and 80 °C) for 45 minutes.

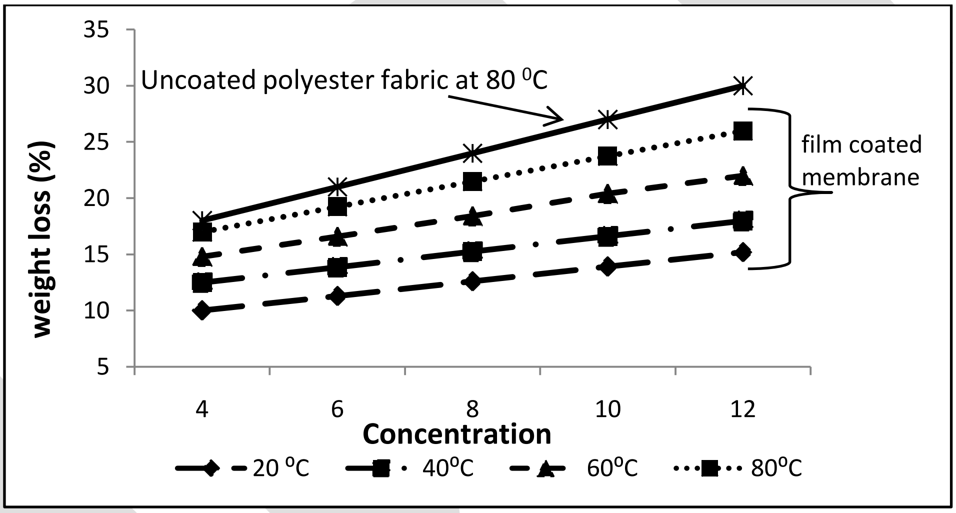


Figure (10): Effect of different concentration of sulfuric acid on weight loss of samples at different temperature (20°C, 40°C, 60°C and 80°C) for 60 minutes.

IV-2.2Effect of acids action on water content (moisture regain):

Figures (11, 12)illustrate the effect of acids on water content of PVA film and PVA film coated polyester membrane. It is clear from **Figures (11,12)** that the PVA coated polyester membrane slightly affected with increasing of acid concentration. But the PVA film showed much higher value of moisture regain in all cases. The results indicate that the moisture regain reveled to the polyvinyl alcohol film, which have high moisture absorbency (Abdulkhani, 2013) and slightly affected by acid. Moreover, the percentage of PVA / polyester after the acids action was increased. The moisture regain will increase according to increase (PVA)/polyester ratio.

Previous reports **(**Kandpal*et al*, 2015;Chuang *et al*, 2002;Brashear, 2001;McIntyre, 1998**)** said the action of sulfuric acid on polyester fabric is increased by increasing its concentration even so at low temperature.

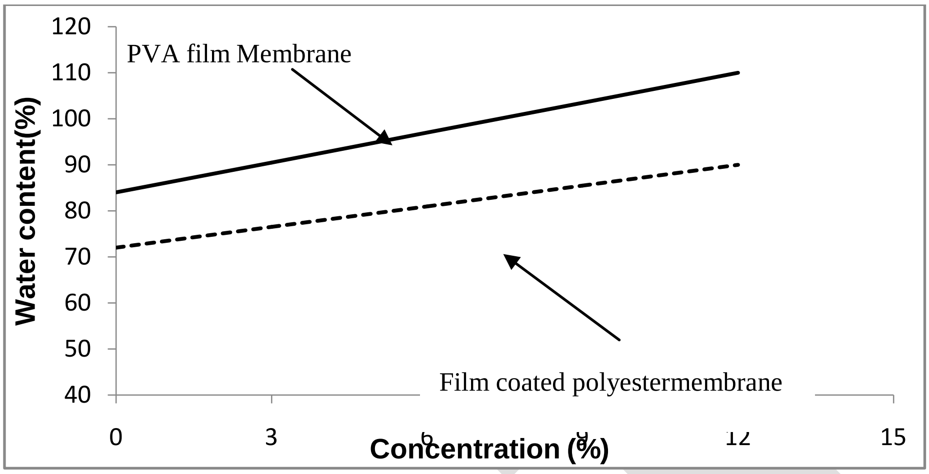


Figure (11): Variation of water content of PVA film membrane and PVA film coated polyester with sulfuric acid concentration in soaking medium.

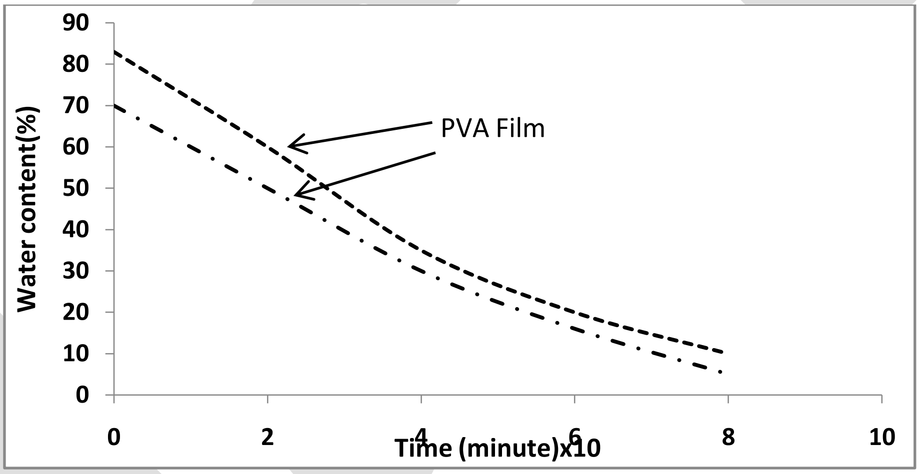


Figure (12): Effect of drying time on water content of PVA film and PVA film coated polyester membrane.

IV-2.3 Effect of inorganic acids on tensile strength:

As it is evident from weight losses by the action of acid. The degradation of film-coated polyester is limited on polyester side only due to the excellent resistivity of (PVA) film coat and it was protect the side of interface between coated film and polyester fabrics, thus the polyester side was affected only. **Figures (13, 14)**represents the tensile strength value of acid affected samples. It is clear that the tensile strength value slightly decreases at low reaction temperature in case of using phosphoric and nitric acids, where the tensile strength value slightly decrease with increasing acid concentration. However, rising the temperature to 80°C with increasing the acid concentration is accompanied with a decrement in tensile strength value in case of using phosphoric and nitric acids. But in case of using sulfuric acid there is a significant decrease in tensile strength value even so at low temperature with increasing the concentration of sulfuric acid as can be seen from**figures (13, 14).**

The observed decrease in the tensile strength value may be due to the strong effect of sulfuric acid which dissolve polyester fabric **(**Kordoghli *et al, 2012* Korshak and Vinogradova, 1965; Densmore, 1960**)** and gives thinner product of film coated polyester membrane.

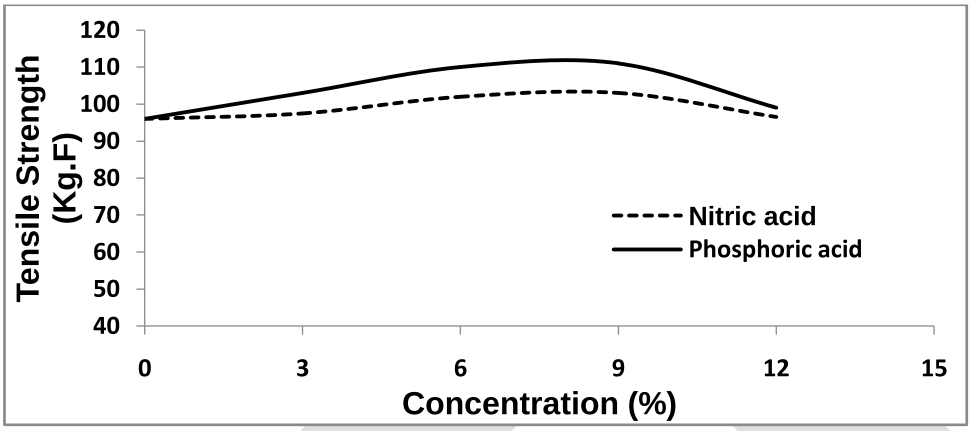


Figure (13): Variation of tensile strength of film coated polyester membrane with phosphoric and nitric acids concentration in soaking medium.

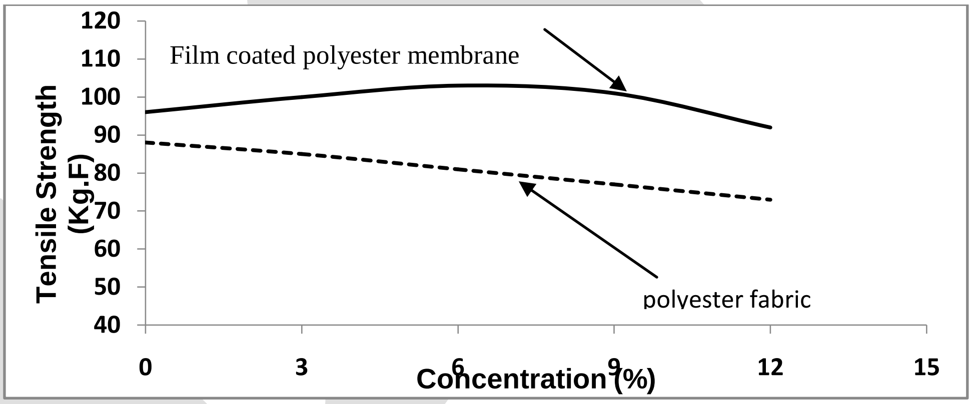


Figure (14): Variation of tensile strength of film coated polyester membrane with sulfuric acid concentration in soaking medium.

IV-2-4 Effect of acid action on the elongation % at break:

Elongation measurement are undertaken to describe changes on the mechanical properties, which happened during the acid action on film coated, and uncoated polyester fabrics.

It is clear from **figures (15)** that the elongation % at break of acid affected samples, increase with increasing acid concentration and time at constant temperature in all kind of acid. But the elongation % at break is much higher in case of using sulfuric acid. This may attributed to the strong effect of sulfuric acid on polyester fabric, which increase the PVA/ Polyester ratio.

Raising the reaction temperature with increasing the acid concentration, causes a higher elongation of the polyester samples.

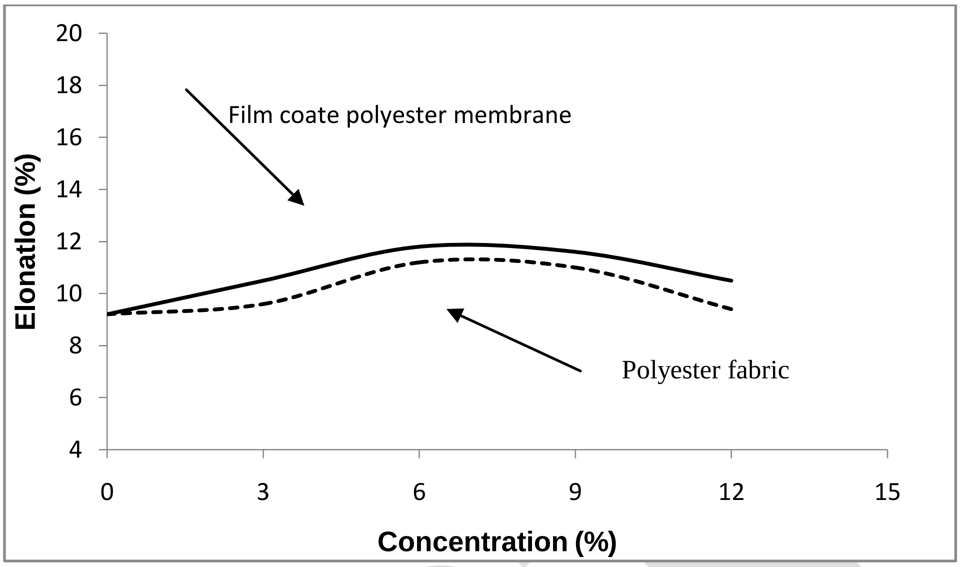


Figure (15): Variation of elongation % at break of coated polyester with deferent concentration of sulfuric acid in soaking medium.

IV-2.5Effect of inorganic acids on abrasion resistant:

The results obtained signify that, the abrasion resistance of samples under test, expressed as a % loss in weight due to abrasion using abrasive paper GRIT 240 for 300 cycle, were decreased by increasing acid concentration at 80 ^{0}C and 1 hour soaking time. Also the effect of sulfuric acid is much higher than the others (Nitric and Phosphoric). **Figure (16)** represent such results.

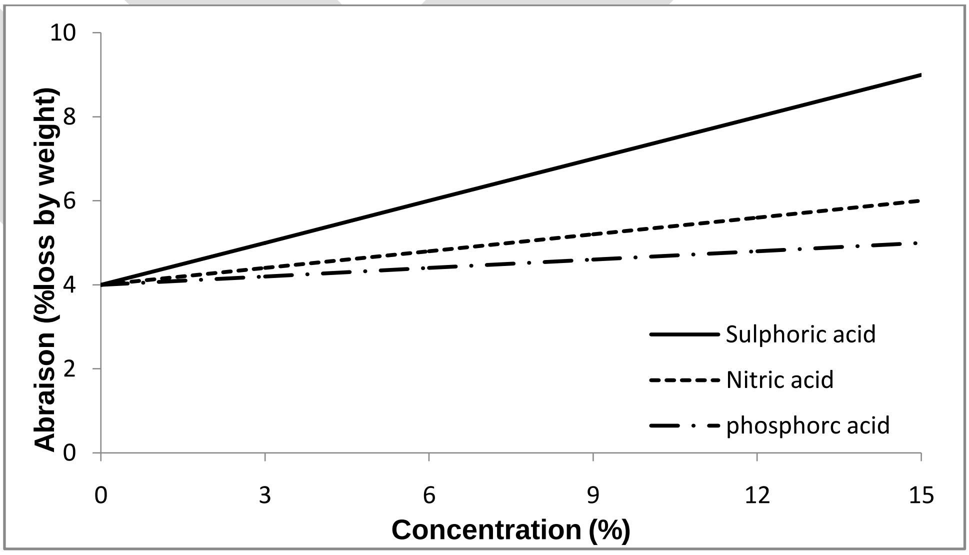
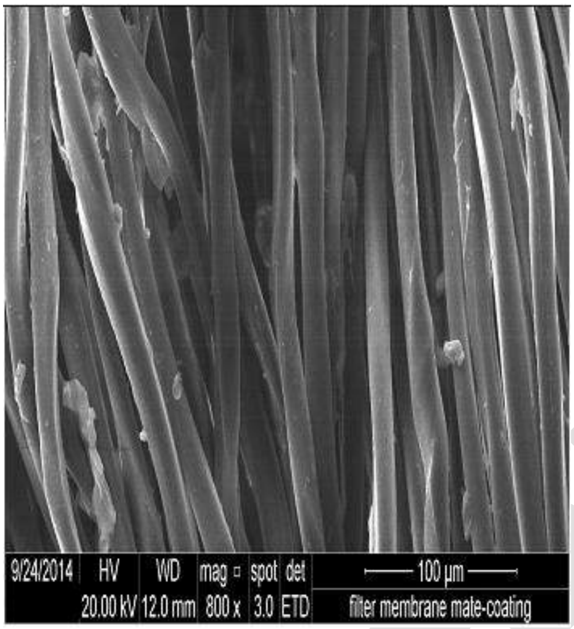


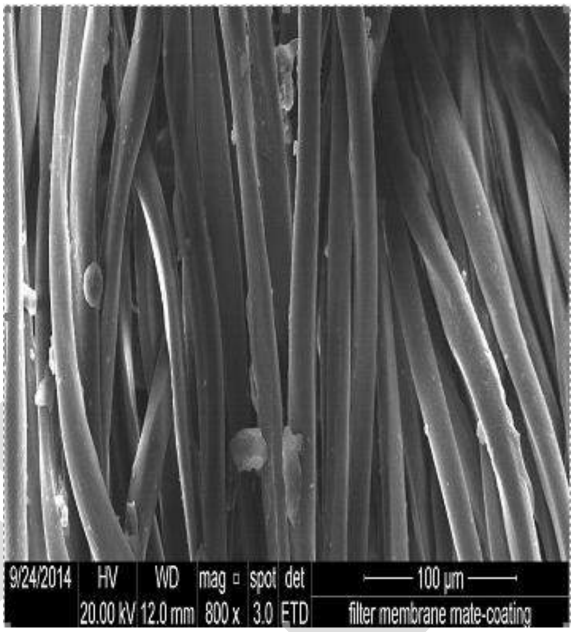
Figure (16): Variation of Abrasion of film coated polyester membrane with deferent concentration of sulfuric, nitric and phosphoric acid in soaking medium at 80 ^{0}C for 1 hour.

IV-2.6 Effect of sulfuric acid on surface morphology of film coated PVA/polyester membrane:

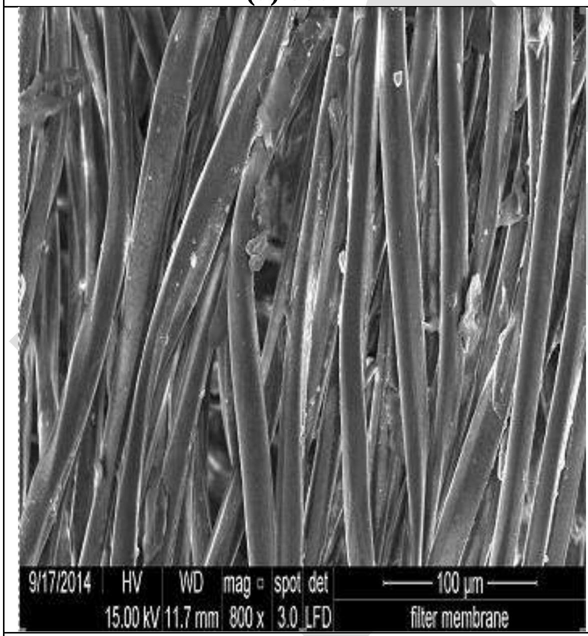
The effect of sulfuric acid on both side of film coated membrane was shown in the following figure (17 , 18)



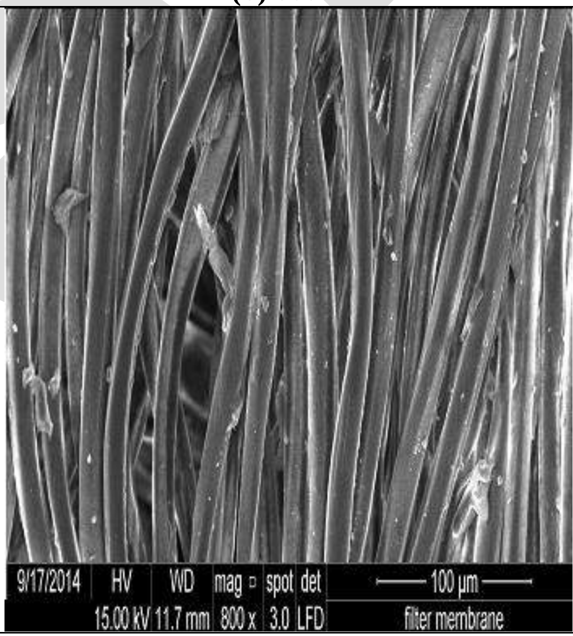
(a) 800 X



(b) 800 X

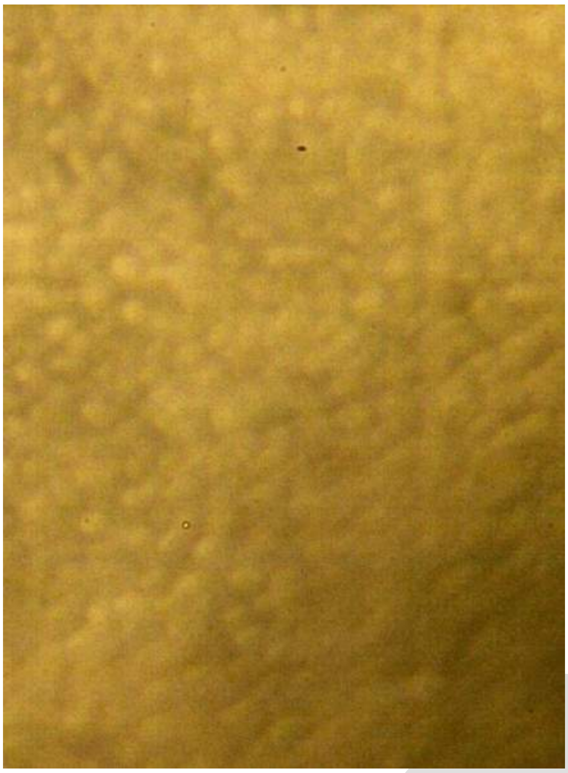


(c) 800 X

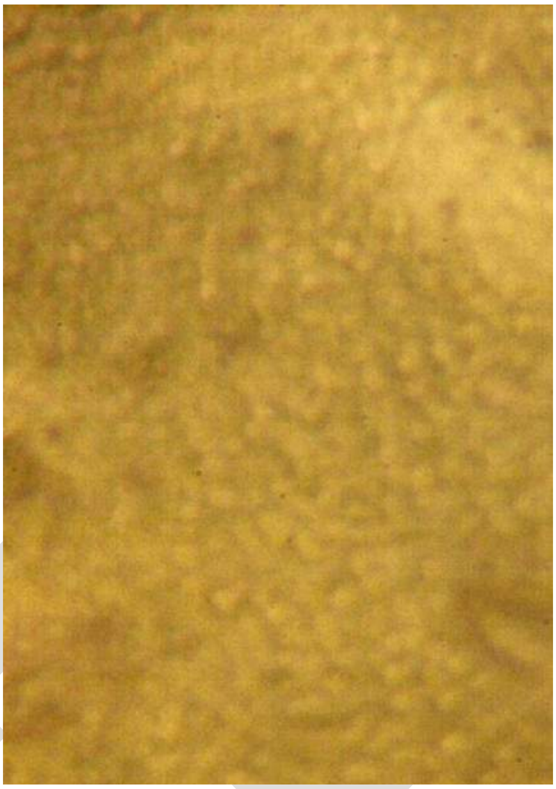


(d) 800 X

Figure (17): Scanning electron microscope (SEM) of PVAfilm coated polyester membrane from the polyester fabric sideaffected by (a- 3%, b-6%, c-9% and d-12%) sulfuric acid at 80^0C for 1 hour.

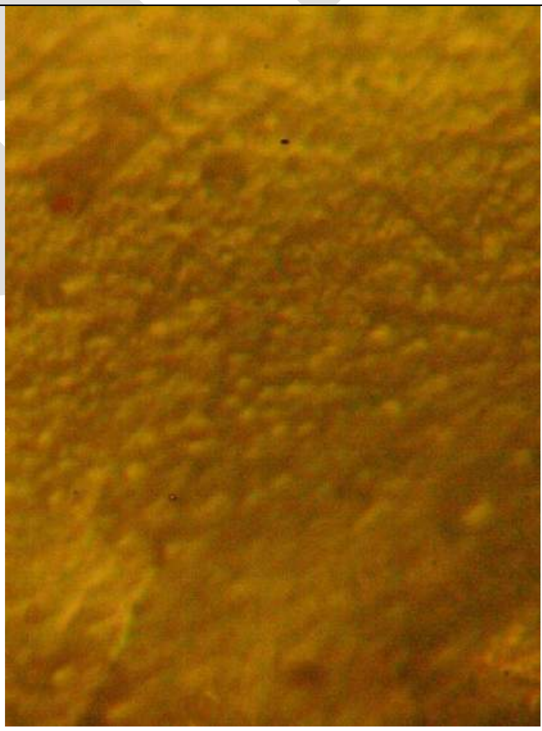


(a) 800 X



(b) 800 X

(c) 800 X



(d) 800 X

Figure (18): Scanning electron microscope (SEM) of PVA film coated polyester membrane from the PVA film side affected by (a- 3%, b-6%, c-9% and d-12%) sulfuric acid at 80^{0}C for 1 hour.

V- REFERENCES:

Abdulkhani, A.; Marvast, E. H.;Ashori, A.;Hamzeh, Y. and Karimi, A. N. (2013). **'Preparation of cellulose/polyvinyl alcohol biocomposite films using1-n-butyl-3-methylimidazolium chloride'**. International Journal of Biological Macromolecules. (62):379–386.

Ashrafa, M. A.; Maaha, M. J.;Qureshia, A. K.; Gharibrezab, M.andYusoffb, I. (2013). **'Synthetic polymer composite membrane for the desalination of saline water'**.Desalination and Water Treatment .51:3650–3661.

Ba, C. (2010). **'Design of advanced reverse osmosis and nanofiltration membranes for water purification'.**Materials Science and Engineering.Urbana-Champaign, University of Illinois.

Brashear, R. L(2001).**'Accelerated degradation of biodegradable poly(ester)s by acidic catalysts'**. University of Iowa.207.

Cerpakovska, D. and Kalnins, M.(2012).**' Barrier and sorption properties of porous poly(vinyl alcohol)–cellulose fibre composites'**.Estonian Academy of Sciences. 61, 3, 178–184.

Chuang, W.Y.; Young, T. H. and Chiu, W. Y. (2000). **'The effect of acetic acid on the structure and filtration properties of Poly (vinyl alcohol) membranes'**. Membrane Science. 172, 241-251.

Das, K.; Ray, D.; Bandyopadhyay, N, R.; Sahoo, S.; Mohantym A. K.andMisra, M. (2010). **'Physico-mechanical properties of the jute micro/nanofibril reinforced starch/polyvinyl alcohol biocomposites films'**. Composites: Part B, 42, 376-381,

Densmore, B. E. (I960).**'The Influence of Sulfuric Acid Hydrolysis on the Lateral Order, Strength, Elongation, and Molecular Chain Length of Two Nylon Filament Fibers'**. The Pennsylvania State University, ProQuest, UMI Dissertations Publishing, 1960. 60021.

El-Aassar, A.-H.M. (2012). **'Polysulfone-Polyvinyl Alcohol Thin Film Nano-Composite Membranes: Synthesis, Characterisation and Application for Desalination of Saline Groundwater'**. Applied Science Research. 8, 3811-3822.

Gohil, J. M.; Bhattacharya, A. and Ray, P. (2006). **'Studies on the Cross-linking of Poly(Vinyl Alcohol)'**.Journal of Polymer Research 13: 161–169.

Hassanien, A. M.; EI-Hashash, M. A.; Guirguis, D. B.and Ramadan, A. M. (2013). **'Fabrication of polyvinyl alcohol/cellulose acetate (PVA/CA/PEG) antibacterial membrane for potential water purification application'**. Hydrology Current Research 4, 146-151.

Kandpal, M.;Sah, D.;Gunwant, V. K. and Upadhyay, A. (2015) **' Application of Acid: For Designing on Cotton Khadi and Khadi Blend Kurties'**.International Journal ofEmerging Research in Management &Technology.(Vol4): 2278-9359.

Kordoghli, B.; Khiari, R.; Mhenni, M. F.; Sakli, F.andBelgacem, M. N. (2012). **'Sulfonating of polyester fabrics by gaseous sulfur oxide activated by UV irradiation'**. Applied Surface Science 258:9737–9741.

Korshak, V. V. andVinogradova, S. V. (1965).**'Polyesters'**.Pergamon Press, Oxford, England.

Loeb, S. and Sourirajan, S. (1963). **'Sea water demineralization by means of an osmotic membrane'**. American Chemical Society, Adv. Chem. Ser., ACS 38. 117–132.

McIntyre, J. E. (1998).**'Polyester Fibres'**. Lewin M, Pearce E M, editor. Handbook of FibreScience and Technology: Fibre Chemistry, Volume IV. NY: M. Dekker, NY, pp 1-73.

Muhammad, R.; Yao, K.; Gong, J.; Chen, X.; Dong-tao, L.; Yi-chao, L.; Dong-mei,C.;Siddiq, M.; and Tang, T. (2015). **'Poly(vinyl alcohol)/GO-MMT Nanocomposites:Preparation, Structure and Properties'**. *Chinese Journal of Polymer Science Vol. 33, No. 2. 329-338.*

Muhammed, A.; Hashash, E.; Mekewi; Guirguis; Ramadan; et al. (2012). **'Polyvinyl Alcohol-Cellulose Acetate Composite Reverses Osmosis Membranes: I. Synthesis and Characterization'.**HydrolCurr Res 3:131.

Rajaeian, B. (2012). **'Synthesis of polymeric nanocomposite membranes for aqueous and non-aqueousmedia'.**M.Phil. Curtin University, Department of Chemical Engineering.

Seyednejad, H.; Ghassemi, A. H.; Van-Nostrum, C.F.; Vermonden, T.andHennink, W.E.(2011).**'Functional aliphatic polyesters for biomedical and pharmaceutical applications'**. J. Control. Release .152, 168–176.

Wang, X.; Fang, D.; Yoon. K.; Benjamin, S. H.andChu,B. (2006).**'High performance ultrafiltration composite membranes based on poly(vinyl alcohol) hydrogel coating on crosslinkednanofibrous poly(vinyl alcohol) scaffold'**. Journal of Membrane Science.278:261–268.