

Effect of graphene oxide on polyvinyl alcohol membrane for textile wastewater treatment

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Abstract. A tremendous amount of energy resources is being wasted in cleaning wastewater to save the environment across the globe. Several different procedures are commercially available to process wastewater. In this work, membrane filtration technique is used to treat the textile wastewater because of its cost effectiveness and low environmental impacts. Mixed Matrix Membrane (MMM) consist of Polyvinyl Alcohol (PVA) in which Graphene Oxide (GO) was added as a filler material. Five different membranes by varying the quantity of GO were prepared. The prepared membrane has been characterized by Scanning Electron Microscopy (SEM), X-Ray Diffractometry (XRD), Fourier Transformed Infrared Spectroscopy (FTIR) and Water Contact Angle (WCA). The prepared membranes have been utilized to treat textile wastewater. The synthesized membranes are used for the elimination of total dissolve solids (TDS), total suspended solids (TSS), Methylene blue (MB) dye and copper metallic ions from textile wastewater. It is concluded that amount of GO has direct correlation with the quality of wastewater treatment. The maximum removal of TDS, TSS, MB and copper ions are found to be 7.42, 23.73, 50.53 and 64.5% respectively and are achieved by 0.02 wt% PVA-GO membrane.

Keywords: graphene oxide; hydrophilicity; PVA membrane; textile; water treatment

1. Introduction

Industrial revolution is leading the world to several different environmental issues, such as but not limited to global warming, shortage of water, disturbance in ecological systems and others (Aryanti *et al.* 2017). All these problems need to be solved for sustainable and healthy life. Among all, the shortage of water is the biggest problem and provision of clean and pure water for local community is one of the major issues of modern world (Haan *et al.* 2018). The problem may even get worse in the coming years, as natural sources for clean water are depleting (Hou *et al.* 2021). In this scenario, the treatment of wastewater or contaminated water to meet the daily water demand is the best possible solution (Cha *et al.* 2020).

The wastewater may be of different types, such as municipal waste, industrial waste, commercial waste, and agricultural waste. Different researchers have characterized the textile wastewater plants and proposed that the treatment of textile wastewater is necessary before discarding to the landfill sites (Rashidi *et al.* 2020).

The treatment of textile wastewater can be done using

oxidation methods, physical methods, and biological methods (Salgot and Folch 2018). Physical methods are considered to be the most effective methods for textile wastewater treatment because of low energy consumption and cost effectiveness. Membrane distillation (MD) is the physical method in which wastewater is treated to produce pure water and for separation of chemicals and is the most cost effective (Calabrò *et al.* 1991). MD is a thermally driven phase separation method which utilizes the hydrophobic membrane for desalination and treatment of wastewater (Alkhudhiri *et al.* 2012). Different researchers have been successfully utilized membranes for treatment of wastewater (Alshahrani *et al.* 2021, Mamah *et al.* 2021, Nawi *et al.* 2020, Qing *et al.* 2020, Roy *et al.* 2020, Tijing *et al.* 2020, Wu *et al.* 2020).

Lau and Ismail (2009) have reviewed different polymeric nanofiltration membranes for the treatment of textile wastewater and summarized that membrane filtration is the most effective method to produce the clean and pure water. Khumalo *et al.* (2019) have used membrane distillation technique for the treatment of water. They studied Polytetrafluoroethylene/Polyvinylidene fluoride (PTFE/PVDF) based membranes and have found that the technique is highly effective in removal of Congo red dye from water, with reported efficiency up to 99%. Bhadra *et al.* (2016) have performed the desalination of water by graphene oxide-based polytetrafluoroethylene (PTFE) membrane and reported the complete salt rejection. Huang *et al.* (2018) have used reduced graphene oxide-based PTFE

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membrane for desalination of water by solar energy and have reported the high salt rejection and clean water production. Laqbaqbi *et al.* (2019) have studied the direct contact membrane distillation technique for the treatment of textile dyes solution by polyvinylidene fluoride (PVDF) membrane and reported the dye absorption on the surface of the membrane. An *et al.* (2017) have used polydimethylsiloxane-based membrane for treatment of textile wastewater and reported complete color removal and complete recovery of pure water. Galiano *et al.* (2018) have developed the polymerizable bi-continuous microemulsion for the antifouling coating of already available membrane and has used for the treatment of textile wastewater and optimized the coating amount based water permeability and dye rejection. Xia *et al.* (2020) have synthesized a starch/polyvinyl alcohol composite and successfully utilized for the treatment of wastewater from textile Srivastava *et al.* (2011) have modified PVDF membranes by styrene-acrylonitrile (SAN) and treated wastewater by varying the composition of SAN as 00:100, 10:90, 20:80, 60:40 and 100:00 and reported that 60:40 is an optimized ratio. It is also observed that all the modified membranes have shown moderate removal of colors and treatment of dyes.

Keeping in view all the discussions, one can conclude that membrane distillation is one of the most effective and efficient process for the treatment of textile wastewater. In the recent past, the use of PVA membranes have gained a lot of interest for wastewater treatment (Amiri *et al.* 2020, Behdarvand *et al.* 2021, Das *et al.* 2020, Ghaffar *et al.* 2019, Ng *et al.* 2020a, Yadav *et al.* 2020). PVA is preferred over other polymeric membranes because of its hydrophilic nature (Huang *et al.* 2021), thermal, mechanical and chemical stability (Yee *et al.* 2014). Gahlot *et al.* (2015) has synthesized PVA-GO membrane but didn't check how PVA-GO membrane will behave for wastewater treatment. Ng *et al.* (2020b) have used PVA-GO membrane for desalination of water and reported that by incorporating GO the desalination was improved by 91.93% but failed to show how much total suspended solvents will be removed. Matrose *et al.* (2019) have successfully removed acid only from rain water by using PVA membrane in which rGO was used as a filler. Guo *et al.* (2019) have also used PVA-GO membrane for forward osmosis application. They have shown that hydrophilicity of PVA membrane is enhanced by incorporating GO in it but they didn't show how much impurities from wastewater can be removed from a PVA-GO membrane. It is clear from literature that PVA-GO membrane is favorable for wastewater treatment but up to the best of authors' knowledge, no one has utilized PVA-based GO membrane for removal of methylene blue dye (MB) and copper ions from textile industry wastewater. In this research, MMM is prepared by using GO as a filler in PVA matrix. GO is considered because of its hydrophilic nature (Tian *et al.* 2019). Membrane is synthesized by solution casting technique and utilized for the treatment of textile wastewater especially for the removal of Cu ions, MB dye, TDS, and TSS. GO is considered as the filler material due to the high strength, light weight and high hydrophilicity. The composition of GO is varied from 0 to 0.02 wt% of PVA to check the impacts of GO nano

particles on the treatment of wastewater. The advantages of the PVA-GO membranes are their easy synthesis method which enables a cost-effective way to remove undesired metallic ions from wastewater.

2. Materials and methods

2.1 Chemicals and reagents

PVA hydrolyzed with MW 89,000-98,000 and GO 2 mg/mL, dispersion in H₂O, Sodium Sulphate and Sodium Hydroxide are taken from Sigma Aldrich. Wastewater is taken from Raza Textile Karachi.

2.2 GO-NPs Membrane synthesis, functionalization and coating

15% PVA solution is prepared by dissolving 15 grams PVA resin in 100 ml distilled water. The mixture is then placed on the hot plate and heated at 90°C for one hour and constantly stirred with magnetic stirrer until transparent solution is obtained. Prior to use for further application, the solution is left to cool down to the room temperature.

The homogenous transparent PVA solution is poured and spread uniformly on glass plate for membrane preparation. The thickness of the membrane was controlled with the help of doctor blade. Then the membrane is allowed to dry for 24 hours at room temperature. Finally, after drying, the casted membrane is separated from the glass plate. The membrane is characterized and utilized for filtration of textile wastewater.

MMM is prepared by addition of GO in casting solution of PVA. Five different membranes by varying the quantity of GO from 0 to 0.02 wt% are prepared by adding GO filler in PVA matrix. 5 mg GO nano powder, 15 grams of PVA resin is mixed in 100 ml distilled water. The mixture is placed on the hot plate and heated at 90°C for one hour and constantly stirred with magnetic stirrer until the translucent solution is obtained. Prior to further using, the solution is cooled down to room temperature. Same procedure is followed for the preparation of 0.01, 0.015 and 0.02 wt% of GO nanoparticle based PVA membrane.

2.3 Membrane characterization

The synthesized membranes are characterized by Fourier Transform Infrared Spectroscopy (FTIR NICOLET IS50), X-Ray Diffraction (XRD Unit Phywe 4.0), Scanning Electron Microscopy (SEM, JEOL JSM-5900) and Water Contact Angle (WCA). FTIR is carried out to explore the functional groups of PVA membrane and PVA-GO membrane. XRD is conducted to evaluate the synthesized membrane crystal structure. SEM is conducted to find out the microstructure of a PVA and PVA-GO membrane. Water Contact Angle (WCA) of PVA and PVA-GO membrane has been carried out to check how hydrophilicity of PVA is affected by addition of GO.

Textile wastewater is obtained from Raza textile Karachi. The characteristics of wastewater provided by

Table 1 Characteristics of untreated wastewater

Impurity	Concentration (mg/L)
TDS	3100
TSS	1180
MB	13.24
Cu ions	0.0955

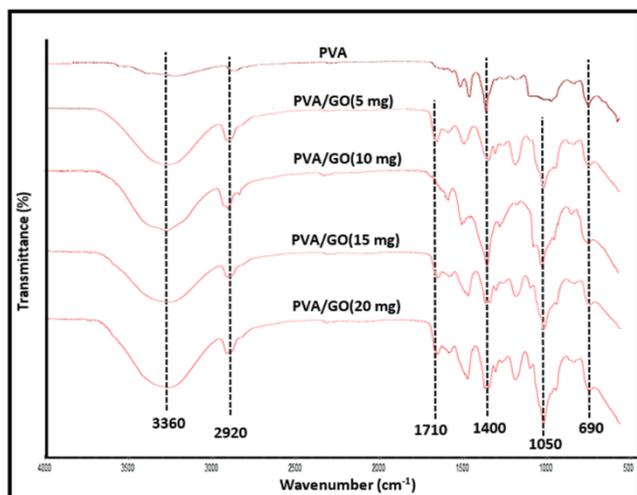


Fig. 1 FTIR curve of PVA and PVA-GO membrane formed

industry is given in Table 1. TDS is measured by means of TDS METER. The concentration of TSS was analyzed by the help of traditional filtration process, via four-micron filter film. Concentration of Copper ions and Methylene blue dyes are measured by the classified setup of Raza Textile Karachi.

3. Results and discussion

3.1 Membrane structure properties

3.1.1 Fourier transform infrared spectroscopy (FTIR)

In general, changes in chemical structure occur while cross linking processes which is studied by FTIR as shown in Fig. 1. The characteristic peak at 690 cm^{-1} recognizes the vibration of C-H aliphatic bending. The peak at 1400 cm^{-1} and 3360 cm^{-1} represents the stretching vibration of hydroxyl functional group (-OH groups). The peak at 2900 cm^{-1} attributed to the stretching of $-\text{CH}_2$ bonds.

In addition, the new peak of wavelength has taken place at 1050 cm^{-1} that indicated the stretching vibration of attributed to the epoxy C-O-C group and the adsorption bands for the carboxyl -COOH group was depicted at 1710 cm^{-1} (Gensterblum *et al.* 2014). These new peaks represent the existence of GO in PVA membrane. Hence the PVA-GO nanocomposite membrane was successfully formed.

3.1.2 X-ray diffraction (XRD)

The XRD pattern of PVA and PVA/GO nano composite membrane is mentioned in Fig. 2. In XRD pattern, a sharp

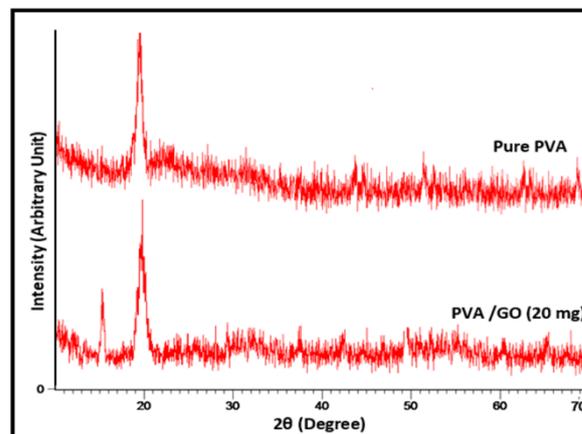


Fig. 2 XRD Curves of PVA and PVA-GO membrane

peak at $2\theta = 19.8^\circ$ corresponds to the semi-crystalline nature of pure PVA membrane. The result of XRD is in good agreement with those available in literature (Tommalieh *et al.* 2021) showing that PVA membrane is successfully synthesized. X-Rays have easily passed the membrane and detected the crystal structure of pure PVA. On the other hand, as mentioned in Fig. 2, the XRD peak of PVA/GO nanocomposite membrane can be seen at $2\theta = 19.8^\circ$ which is again showing the presence of PVA in membrane while a small peak at $2\theta = 10.5^\circ$ is showing the presence of GO filler (Johra *et al.* 2014) in the PVA/GO MMM and it specifies that the GO is uniformly and homogeneously dispersed in the polymer PVA matrix.

3.1.3 Membrane morphology

SEM study is conducted to find out the microstructure of a PVA membrane and PVA /GO (20 mg) composite membrane. The results are in good agreement with study conducted by Falath (2021) who have used GO based PVA membrane for reverse osmosis. Figs. 3(a) and 3(b) demonstrates the PVA membrane surface and PVA/GO nanocomposite membrane surface, respectively. Both of which are resulted in the form of dense membrane structure. The visible variations between the surface pictures of the pure PVA membrane indicates a clean surface and a smooth morphology relative to the PVA/GO membrane, which has a rougher surface with homogeneous strip structures and is denser due to GO dispersion in the PVA matrix. It is shown that GO is exfoliated effectively and spread quite uniformly in the PVA matrix. The improvements in the membrane structure of the PVA membrane to the PVA/GO nanocomposite membrane is observed more easily and distinctly from the cross-sectional picture of SEM of both the membranes. Figs. 3(c) and 3(d) indicates the cross-sectional SEM picture of pure PVA membrane and PVA/GO membrane, respectively. The cross section of the PVA/GO nanocomposite membrane is noticeably dissimilar from that of the pure PVA membrane. The PVA/GO film is grainier, and the structure is more regulated. A loading of GO results in the creation of strong van der Waals interactions between GO layers. Furthermore, the composite film surface shows strong homogeneity, which suggests the integration of GO in the PVA matrix. Owing to the small

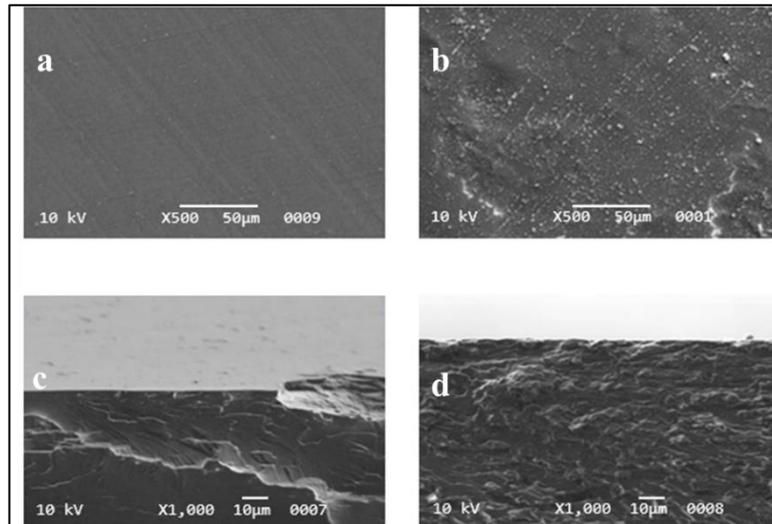


Fig. 3 (a) SEM image of PVA membrane, (b) SEM image of 20 mg PVA-GO membrane, (c) Cross Section of PVA membrane, (d) Cross Section of 20 mg PVA-GO membrane

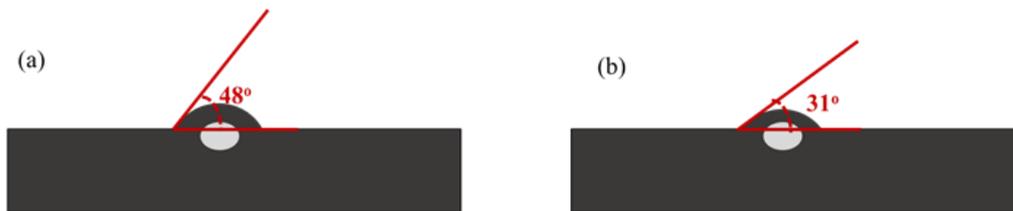


Fig. 4 WCA of (a) pure PVA, (b) 0.02% PVA-GO

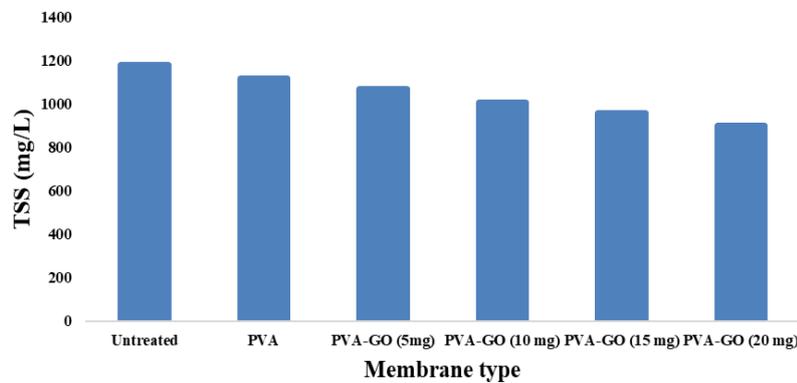


Fig. 5 TDS of wastewater after treatment

cluster size and GO boundary in the composite membrane this arrangement should limit the permeability of pollutants as GO is well distributed in the PVA polymer matrix.

3.1.4 Membrane hydrophilicity

Fig. 4 shows the water contact angle of pure PVA and 0.02% PVA-GO membrane. The pure PVA membrane is showing WCA of 48° (Fig. 4(a)), which means that PVA is hydrophilic in nature and is an effective option for water treatment. By the addition of GO filler in PVA, WCA is decreased to 31° . The decrement in WCA is indicating that by adding GO in PVA, hydrophilicity is enhanced due to hydrophilic nature of GO. This enables hydrophilic PVA membrane to transform into super hydrophilic membrane as also discussed by Li *et al.* (2019). The hydrophilicity is

enhanced because addition of GO in PVA increases the pore size and allow more permeation of water. Also, it makes PVA-GO membrane to be more attractive option than pure PVA membrane for treatment of water.

3.2 Transmembrane water permeation and species rejection

TDS of untreated and treated water from PVA membranes with different loading have been measured and results are presented in Fig. 5, from where it is evident that TDS of un-treated textile wastewater was 3100 mg/L. The addition of GO in PVA favors in rejection of TDS and is highly favorable for treatment of wastewater. TDS of water filtered through pure PVA membrane is measured as 3060

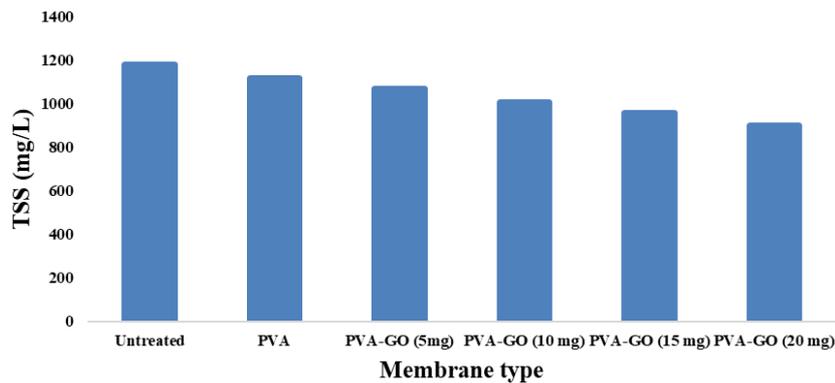


Fig. 6 TSS of wastewater after treatment

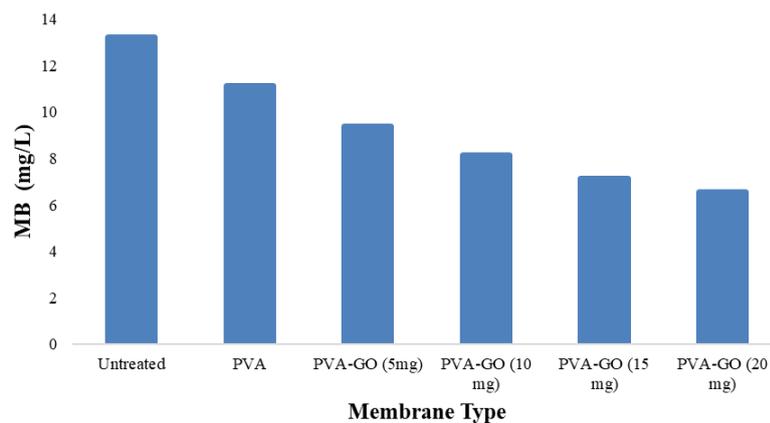


Fig. 7 MB amount of untreated and treated wastewater

mg/L that indicates the removal efficiency of 1.29%. TDS of water filtered through PVA- GO (5 mg) membrane was measured as 3000 mg/L which increases the removal efficiency up to 3.22%. Furthermore, the TDS of water filtered through PVA- GO (10 mg) membrane is measured as 2950 mg/L and the removal efficiency is indicated as 4.83%. TDS of water filtered through PVA- GO (15 mg) membrane is measured as 2910 mg/L that indicates the removal efficiency of 6.12% and the TDS of water filtered through PVA- GO (20 mg) membrane is measured as 2870 mg/L that indicates the removal efficiency of 7.42%. By increasing the GO nanoparticles in PVA membrane, more TDS can be removed. The addition of GO in PVA matrix causing the hindrances in passing of TDS through the membrane structure. Thus, more TDS are blocked by increasing the amount of GO filler in PVA matrix.

When discarding the wastewater, it is important to remove TSS from it. As that of TDS, TSS of untreated and treated wastewater are also measured and similar trend is observed in TSS rejection. The impacts of PVA-GO membrane on the removal of TSS is presented in Fig. 6, it is noticed that the TSS of untreated textile wastewater is 1180 mg/L. The Pure PVA membrane minimized the total suspended solids to 1120 mg/L, which indicates the removal efficiency of 5.08%. TSS of water filtered through PVA-GO (5 mg) membrane is measured as 1070 mg/L that indicates the removal efficiency of 9.32%. Also, the TSS of water filtered through PVA-GO (10 mg) is measured as 1010 mg/L that indicates the removal efficiency of 14.4%.

TSS of water filtered through PVA- GO (15 mg) membrane is measured as 960 mg/L, representing a removal efficiency of 18.64% and the TSS of water filtered through PVA-GO (20 mg) membrane is measured as 900 mg/L that indicates the removal efficiency of 23.73%. It means by treating water through membrane more and more TSS can be eliminated. By addition of GO in PVA matrix, a resistance for the flow of TSS was offered by GO filler thus providing clean water and filtrate was found to be free of TSS.

The MB of treated and untreated wastewater are also determined and it has been found that addition of GO in PVA is favorable for MB removal. Fig. 7 shows the amount of MB in untreated and treated wastewater through different membranes. From Fig. 7, it is observed that the concentration of MB dye in un-treated textile wastewater is 13.24 mg/L. MB of water filtered through pure PVA membrane is measured as 11.14 mg/L that indicates the MB removal efficiency of 15.86%. MB of water filtered through PVA- GO (5 mg) membrane is measured as 9.38 mg/L that indicates the MB removal efficiency of 29.15%. Furthermore, the MB of water filtered through PVA- GO (10 mg) membrane is measured as 8.14 mg/L that indicates the MB removal efficiency of 38.52%. MB of water filtered through PVA- GO (15 mg) membrane is measured as 7.13 mg/L that represented the MB removal efficiency of 46.14% and the MB of water filtered through PVA- GO (20 mg) membrane is measured as 6.55 mg/L that shown the MB removal efficiency of 50.53%.

As more TDS, TSS and MB are rejected by increasing the loading of GO in PVA. Cu ions are also found to be

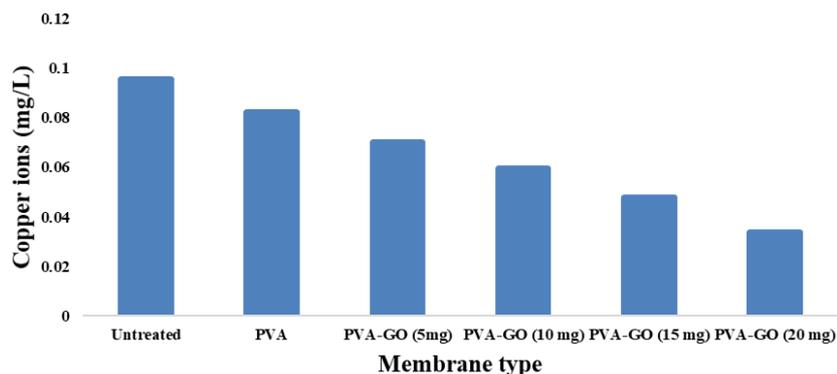


Fig. 8 Copper ions amount of untreated and treated wastewater

rejected more by increasing the loading of GO in PVA. Fig. 8 shows the amount of copper ions in untreated and treated wastewater. From Fig. 8 it is found that the concentration of Cu ions in un-treated textile wastewater is 0.0955 mg/L. Cu ions of water filtered through pure PVA membrane is measured as 0.0825 mg/ L, indicating a Cu ions removal efficiency of 13.61%. Cu ion of water filtered through PVA- GO (5 mg) membrane is measured as 0.0703 mg/ L that shows the removal efficiency of 26.38%. Furthermore, Cu ions of water filtered through PVA- GO (10 mg) membrane is measured as 0.0598 mg/L that suggests the removal efficiency of 37.38%. Cu ions of water filtered through PVA- GO (15 mg) membrane is measured as 0.0478 mg/ L, indicating a Cu ions removal efficiency of 49.94%. The Cu ions of water filtered through PVA- GO (20 mg) membrane is measured as 0.0339 mg/L that represents the removal efficiency of 64.5%.

4. Conclusions

The textile wastewater is selected for the study. PVA and PVA-GO membrane is used for the treatment of textile wastewater. Five different membranes with different concentrations of GO are prepared and used for the treatment of textile wastewater. The prepared membranes are characterized by FTIR, XRD, SEM and WCA. From the analysis, one can conclude that GO is uniformly distributed in the membranes prepared. Also, addition of GO in PVA is an attractive option for enhancement of hydrophilicity. The membranes prepared are used for the removal of TDS, TSS, MB and Cu ions from the textile wastewater. The PVA- GO based membrane is found to be more efficient and effective than pure PVA in removal of TDS, TSS, MB and copper ions. The quantity of GO is varied from 5 mg to 20 mg. The maximum removal of TDS, TSS, MB and copper ions are found to be 7.42%, 23.73%, 50.53% and 64.5% respectively and are achieved by 0.02wt % PVA-GO membrane. By increasing the amount of GO nano particles more removal of TDS, TSS, MB and Copper ions can be achieved.

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