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DEVELOPMENT OF HDPE-GO COMPOSITE MEMBRANES FOR EXTENUATING THE TOXIC RELEASE OF CHROMIUM METAL FROM LEATHER INDUSTRIES

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ABSTRACT

The effluent produced by the leather industries constitutes a potent waste combination. Prior attempts to mitigate environmental factors and treat wastewater involved measures with widespread adverse repercussions. This research focuses on the fabrication of a composite membrane through thermal-induced phase separation (TIPS) regulated by hydrogen bonding interactions within GO's OH-group and HDPE's C-H group. FTIR validates the binding between GO and HDPE, while FESEM evaluates the topographical structure. Contact angle recordings demonstrate the membrane's hydrophilic. For optimal water flow and wastewater rejection, HDPE-GO composite solution volumes are optimized to adjust membrane composition. The membrane was applied to remove carcinogenic chromium metal from post tanning wastewater through an permeation mechanism. Membrane performance was assessed in dead-end filtering systems. The 0.20% GO in HDPE composite membrane exhibits 8.56 MPa tensile strength, 15% elongation at break. With good wastewater flux, the highest pure water permeability was 52 $Lm^{-2}h^{-1}$. In performance evaluation, chromium metal rejection was significant with 85% and improved pure water flux from 15 to 52 $Lm^{-2}h^{-1}$. Higher hydrophilicity, better mechanical properties and improved metal rejection capabilities were found in the developed graphene-enhanced HDPE membrane. Compared to conventional HDPE membranes, the addition of graphene significantly increased flow and rejection rates for chromium and metal complex dyes. These findings underscore the efficacy of HDPE-GO composite membrane in TDS separation and removal of other wastewater contaminants.

Keywords: *Membrane composite, Wastewater Treatment, Filtration, Pollutants Separation*

1. INTRODUCTION

Man-made disasters referred to as an anthropogenic hazard, arises due to human actions or inactions possessing significant responsibility to inflict injuries and suffer to humans, plants and animals. Besides, anthropogenic hazard is harmful to the whole existence such as lands, forest, oceans etc. (Maskuriy, 2020). On earth, all industry stakeholders such as manufacturing, farming, construction have a great stress on the effects of the human made hazard for every single living thing. It encompasses technological hazard such as fire explosions, leakages, toxic releases, structural collapses of physical infrastructure, and various forms of pollutions such as water, air, soil, acid rain, etc. (Shaluf, 2007). Water pollution is an anthropogenic hazard that is mostly occurred by industrial wastewater. Among different kinds of industry, leather-processing industry named tannery is considered to be the most contaminating source for production of wastewater. Despite the fact, there is no hard evidence about the water pollution caused disaster over the world. Nevertheless, many researcher believes that a large amount of toxic chemicals discharged to the waterbody leads to damage environment for making different kinds of problems as hazard which might contribute in the increasing the global pollution (Karri, 2021).

According to the World Bank, tannery wastewater accounts for up to 50% of the total industrial pollution in developing countries. On a global scale, approximately 600 million m^3 of wastewater is released into water bodies annually through the processing of 17 million tons of hides and skins (Rajamani, 2017). In Bangladesh, more than 200 tannery industries release 20000 m^3 of wastewater per day (Saha, 2021). UNIDO has identified the involvement of 175 chemical types in tannery operations, a factor that has resulted in significant environmental repercussions when effluents are inadequately treated (Ricky et al., 2022). Among these chemicals, the basic chromium sulfate and metal complex azo dyes are being applied as tanning agent and dyeing agent, which are most important chemicals in leather processing operation. As a result, these toxic chemicals are being discharged as wastewater into the water body. Chromium is a known carcinogen, which contaminates soil and water sources leading to ecosystem disruption, threating to health of human. (Juel et al. 2022; Georgaki et al., 2023). Additionally, these ugly wastewater confer various health hazard in human body such as tissue necrosis, organ damage, jaundice, hypertension, hemolysis, respiratory etc. (Kannaujiya et al., 2021).

Across the globe, communities have encountered a rising occurrence of disasters, stemming from diverse and multifaceted origins, leading to both direct and indirect consequences. Among the influential cause-and-effect factors, the environment stands out as a pivotal element. The world has already faced a several anthropogenic disaster in history such as love canal, minamata disaster due to dumping chemical waste into land, releasing methylmercury into the local waterbody respectively (Parker, 2013). While significant emphasis has been placed on understanding the detrimental aftermath on the environment occurred by the disaster incidents, relatively limited awareness has been dedicated to comprehending ramifications of the inadequate management practices of environment and ecological deterioration that can exacerbate the severity of disasters (Srinivas et al., 2008). Therefore, addressing these issues necessitates stringent regulations, waste management strategies, and the adoption of eco-friendly alternatives in the leather industry.

Recently, wastewater treatment is going through membrane technology, which is one of the most favored methods. Organic membranes encompass petroleum-derived synthetic matrix (polymers) such as polyether sulfone (PES), polysulfone (PS), polyvinylidene fluoride (PVDF). Besides, inorganic derived membranes encompass carbon molecular sieves, ceramics, amorphous silica, zeolites a variety of other materials (Dong et al., 2021). Membrane technology yields the potential to give attention to the gap between sustainability and economic viability by enabling the reduction or elimination of chemical usage, promoting environmental friendliness, and ensuring accessibility.

High-density polyethylene (HDPE) is a prevalent plastic frequently employed for packaging applications. Furthermore, commercial HDPE has gained substantial traction in membrane manufacturing, showcasing notable effectiveness in water treatment, evidenced by an impressive 89.54% humic acid rejection rate and a flux of 5 $Lm^{-2}h^{-1}$. In the context of this study, HDPE was utilized as the fundamental material for wastewater treatment membranes. A parallel approach involving the use of HDPE for membrane fabrication was previously reported by Zulfiani et al., Zukimin et al. (Zulfiani et al., 2023). There has been a concentrated endeavor to enhance polymer membranes by integrating inorganic nanoparticles (such as SiO2, TiO2, ZrO2, ZnO, Al2O3, CNTs, GO) in suitable quantities leads to a noteworthy enhancement in both hydrophilicity and permeability of the resultant membranes, achieved through structural modifications. Currently, graphene oxide (GO) boasts an abundance of hydrophilic oxygen functional groups, two-dimensional nanoplate structure, exceptional mechanical properties, significant specific surface area on both its basal planes and edges (Xing et al., 2015). Hence, it is now an appealing candidate for the design of advanced membranes across a spectrum of disciplines, including wastewater treatment. The application of GO nanosheets has been found to lead to a lessening pore diameter on the surface with a more refined distribution of pore sizes within nanofiltration membranes (Kazemi et al., 2021).

Although research on the use of HDPE and GO separately in membrane technology is available, the performance of the HDPE composite membrane reinforced with GO have yet to be analyzed. This research aims at developing a composite HDPE-GO membrane for extenuating the toxic release of chromium in the aquatic environment. The developed membrane was characterized and analyzed through FTIR, SEM, and tensile test, respectively to ensure bonding, morphological and strengths. Furthermore, the extenuating efficiency of the membrane was optimized for the release of toxic chromium from tannery wastewater. Thus, a prevention approach for human-made disaster management has been devised in this study to alleviate the potential risks of toxic release in the aquatic environment.

2. EXPERIMENTAL

2.1 Materials

The matrix material, which is HDPE, was collected from local shop. Mineral oil (Smart Lab, local distributors, Khulna) was purchased for extraction. Tannery wastewater was collected for contaminants treatment from local tannery named SAF Leather Industries Ltd. Noapara, Jashore, Bangladesh.

2.2 Design and fabrication HDPE-GO composite membranes

Via modified Hummer's method graphene oxide (GO) was prepared from natural graphite flakes in laboratory. [Uddin et al., 2015] .Four different membranes were fabricated using thermally induced phase separation (TIPS). First, the prepared GO was dispersed in DMF and sonicated for almost 2 h. Afterward, The HDPE granules was mixed with GO dispersed solvents the aid of a magnetic bit stirrer at 500 rpm for 12 h at 80 °C to attain homogeneity in the dope solution. After preparing the GO, five different membranes were developed through TIPS (thermally induced phase separation). First, the prepared GO was dispersed in mineral oil with sonication for 2 hours. Then, the granules HDPE was taken to this mixer and at 140 C temperature this mixer was homogenized with rotation of 100 rpm for 2 hours. After stirring, this solution was let to have rest for removing the gases. (Zulfiani et al., 2023). On a preheated glass sheet, the homogenous solution was poured to be casted and this was immediately immersed into water bath for phase separation. After peeling, the casted sheet of the developed membranes were immersed in acetone solution for 24 hours for extracting mineral oil. Then these membranes were dried at room temperature. In table 1, the different composition of developed membranes are presented.

2.3 Membrane Characterization:

To ascertain the chemical structure of both GO and one composite membrane, Fourier transform infrared spectroscopy (FTIR) analysis was conducted to get characteristics peak. Through the KBr pellet method, a tensor 27 FTIR spectrometer (bruker, Germany) was employed with span a wavelength range 400 to 4000 cm-1 for analysis. Using Hitachi SU5000 instruments, surface morphology of the neat HDPE and GO incorporated HDPE wereexamined through Field Emission Scanning Electron Microscope (FESEM) with accelerating voltage 20.0 kV. For measuring contact angle, a 3D optical microscope (3D OM, vhx-5000) was utilized to determine hydrophobicity with probing solvent (3L deionized) at room temperature.

Table 1: Compositions of casting solutions

Sample	Membranes	HDPE (%)	GO (%)	Solvents (%)
M_1	Neat HDPE	15.00	0.00	85.00
M_2	HDPE/GO	15.00	0.05	84.95
M_3	HDPE/GO	15.00	0.10	84.90
M_4	HDPE/GO	15.00	0.15	84.85
M_5	HDPE/GO	15.00	0.20	84.80

Using Universal Testing Machine (25st, Tinius Olsen), the mechanical properties of developed composites membranes were evaluated. The tensile strength was found by equation (1)

$$\tau = \frac{F_{max}}{A} \tag{1}$$

Here, τ, F_{max} and A denotes the tensile strength in MPa, maximum strength load and area of membrane respectively. (Zulfiani et al., 2023).

Concurrently, the elongation at the point of fracture was computed using equation (2), wherein Ɛ represents the elongation (expressed as a percentage), d signifies the length at the breaking point (measured in millimeters), and a denotes the initial length (also in millimeters):

$$\mathcal{E} = \frac{(d-a)}{a} \times 100\% \tag{2}$$

2.4 Performance analysis of fabricated HDPE-GO composites membrane:

The chromium and metal-complex azo dyes rejection capacity of manufactured membrane was evaluated with area of 50 cm^2 using a vacuum system, which is also known as dead end filtration. To minimize the effect of membrane compaction on membrane capacity, the prepared membranes were processed using deionized water for 15 mimutes before permeance testing (Zulfiani et al., 2023). The membrane permeability was analyzed using the equation below.

$$J = \frac{V}{(A \times t \times P)} \tag{3}$$

Here, J, V, A, P and t signifies respectively permeate flow rate, permeate volume, membrane surface are, transmembrane pressure and time.

During the experiments, the following equation was used to evaluate the chromium and dyes rejection capability C_p

$$R(\%) = 100 \times (1 - \frac{C_p}{C_F}) \tag{4}$$

Here, R, C_P and C_F represents chromium compound rejection, permeate concentration and feed concentration of chromium compound in wastewater respectively. The concentration of Chromium compound was measured by ICPMS 2030 LS.

3. RESULTS & DISCUSSIONS

3.1 FESEM Observation

Two samples such as M_1 and M_5 was carried out for FESEM test for morphology observation. Nil GO and 0.20% GO incorporation in the developed membranes respectively M_1 and M_5 are shown in Figure 01.

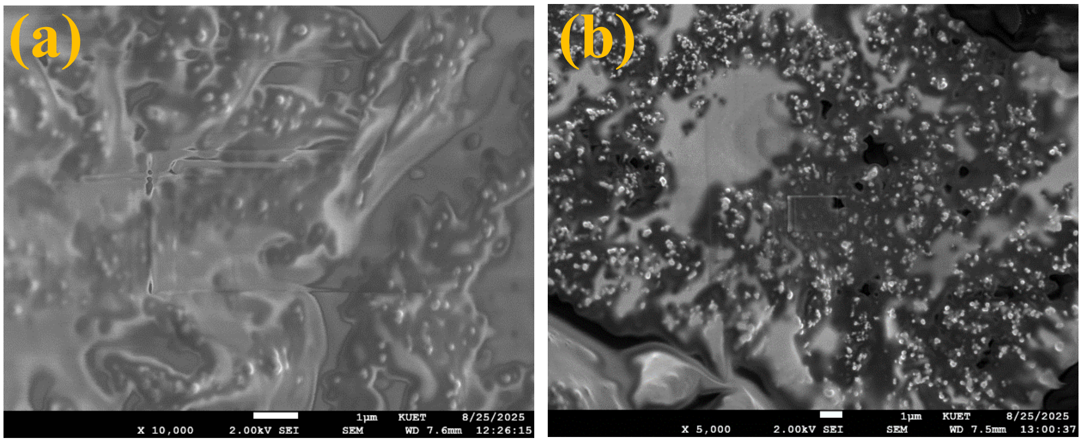

Figure 1: FESEM images of HDPE membrane M_1 (a) and M_5 (0.20%) incorporated HDPE membrane

From this, the effect of GO on the structure of synthesized membranes can be easily observed. Generally, both thermodynamic and kinetic properties of the membranes can be effected through incorporation of GO even at the addition of small amounts (Garcia-Ivars et al., 2015). From figure, it is seen that rougher surface was created on the M_5 membrane than the M_1 which has zero GO. Besides, it is confirmed from images about the cellular pore like structure of developed GO incorporates membrane (Saljoughi et al., 2009). The reason behinds this is the attribution of GO that has great affinity to water molecules resulting the formation of cellular pore like structure in the prepared membranes. As well, the addition of GO has a role to mass transfer of mineral oil and water as solvent and non- solvent materials for the growth of macro void in the layer of membranes. This finding is also found in different studies with different nanoparticles such as Ag, ZnO, SiO_2, TiO_2 etc (Sotto et al., 2011; Balta et al., 2012; Lin et al., 2016). Therefore, it is clear to state that GO presence in membranes has impacts on formation process and structure.

3.2 FTIR analysis

Membrane M_1 and M_5 were tested for FTIR spectra to analysis the interaction among functional groups of GO and HDPE. From figure 02, three characteristics peaks are shown in which stretching vibrations CH_2 at 2912-2840 cm^{-1}, bending vibrations CH_2 at 1380-1360 cm^{-1} and rocking vibrations of CH_2 at 716 cm^{-1}

are prominent in M_1 but some changes of these peaks with addition some new peaks of the spectra of M5 membrane were observed clearly (Balaji Ayyanar et al. 2020; Sarker et al. 2011; Zukimin et al., 2016; Lin et al., 2015). In GO blended M_5 membrane, there are increased absorption at 1305, 1090, 920, 880 cm^{-1} which are respectively for C-H bending vibration. A peak with 1090-880 cm^{-1} was identified for stretching vibration of C-O group as well as at 3441, slightly increased absorption intensity peaks is seen for O-H stretching vibration. The stretch vibration of CH_2 and C-H group was represented on the composition of GO and HDPE materials of the developed GO incorporated Membrane M_5 as the evident absorption bands in 2930-2825 cm^{-1} and 1090-880 cm^{-1}. Furthermore, incorporation of the OH group through GO addition has affected on the improvement of hydrophilicity of developed membranes (Kumari et al., 2020; Zhao et al., 2016; Kazemi et al., 2021; Dey et al., 2023).

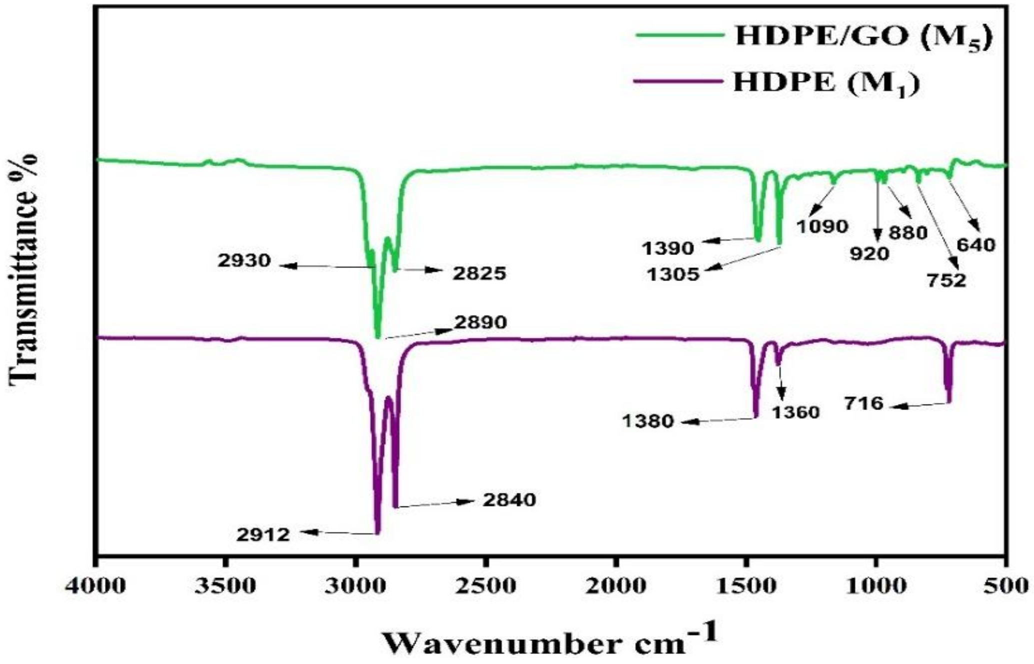

Figure 2: FTIR spectra of developed HDPE (M_1) and GO-HDPE (M_5) (0.20% GO) membranes

3.3 Contact angle analysis

From figure 03, the contact angle, which determines hydrophobicity or hydrophilicity properties of the membranes are shown of the fabricated membranes. The lower contact angle means the lower hydrophobicity i.e. the higher hydrophilicity and vice-versa. The water contact angle were decreasing according to addition of GO content in membranes. In M_1 membrane the contact angle was 108 but in M_5 membrane this was decreased to 70 which indicates the great enhancement of hydrophilicity of membranes due to incorporation of GO. The upgradation of contact angle is directly connected to roughness of the membrane surfaces that causes the hydrophilicity properties of the developed membranes. This is occurred for having large number of hydrophilic functional groups such as OH, COOH that enhances the attraction to the water molecules similarly reported in the previous studies for incorporation of GO nanomaterials (Balaji Ayyanar et al., 2020; Sarker et al., 2011; Zukimin et al., 2017; Lin et al. 2015).

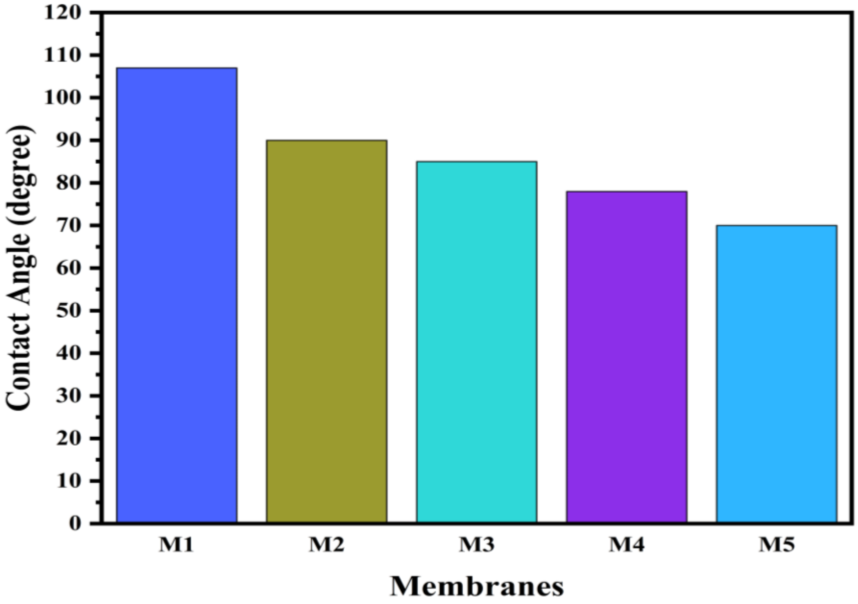

Figure 3: Contact angle of developed membranes

3.4 Mechanical Characterizations

The tensile strength and elongation of developed membranes are shown in figure 04. The tensile strength of membranes were increased after incorporation of GO from 0.58 to 8.56 MPa. M_1 membrane which had no GO, shows 0.58 MPa tensile strength and elongation 2%. When the addition of GO was started, the tensile strength was also increased such as 2.43 MPa for M_2, 4.92 MPa for M3, 8.56 MPa for M_4, 8.27 MPa for M_5.

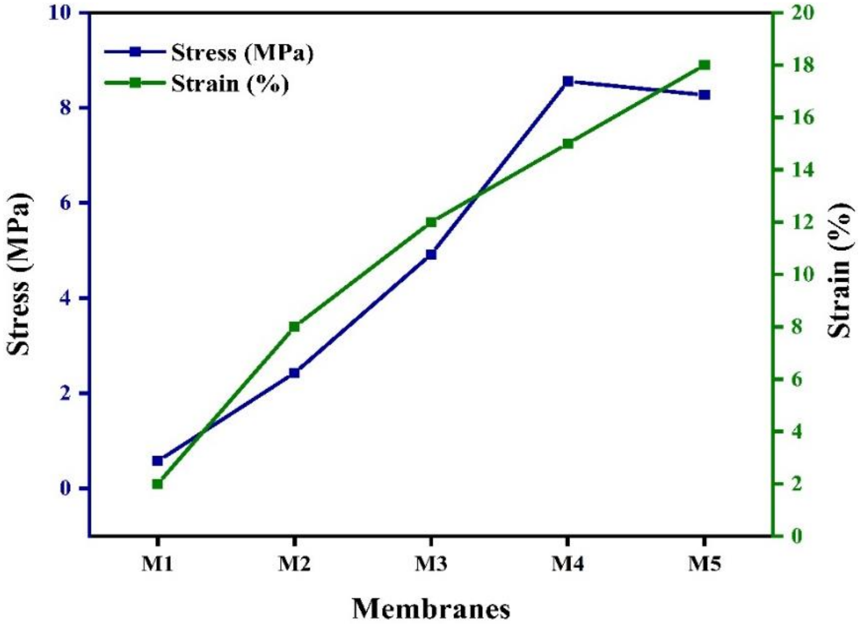

Figure 4: Mechanical Characteristics of developed membranes

This states that GO confers the additional mechanical strength to the membranes with excellent compatibility in HDPE polymer matrix. However, there is a another issue about aggregation of GO when the higher amount was used in this studies such as 0.20% GO (Dey et al., 2023). From the graph, it is seen that 0.15% GO incorporation in HDPE matrix membrane had higher tensile strength 8.56 MPa with elongation 15%. In practical application of wastewater treatment, an excellent mechanical stability is prerequisite for sustaining and backwashing of membranes. In this case, M_4 membrane has good mechanical properties after optimal concentration of GO addition, which is 0.15%.

3.5 Membrane Performance test through flux and Chrome rejection

The fabricated membranes were examined for performance including water flux, wastewater flux and the rejection, which are shown in figure 05. With increasing GO content in composition, the performance were enhanced remarkably with to some extent sacrificing the rejection for increasing hydrophilicity. The M_1 membrane had 15 $Lm^{-2}h^{-1}$ water flux, 12 $Lm^{-2}h^{-1}$ wastewater flux with 58% chrome rejection. After adding GO, the performance were enhanced such as M_2 had 20 $Lm^{-2}h^{-1}$ water flux, 10 $Lm^{-2}h^{-1}$ wastewater flux with 71% chrome rejection. Thus, the performance had been promoted until the GO incorporation had been saturated in the composition. Specifically, the M_4 loaded with 0.15% shows the water flux of 50 $Lm^{-2}h^{-1}$ with 84% chrome rejection. After that, GO showed lower impact on membrane than M_4 membrane. When the GO was increased to 0.20%, the performance of membrane was leveled off with 52 $Lm^{-2}h^{-1}$ water flux, 6 $Lm^{-2}h^{-1}$ wastewater flux with 85% chrome rejection. Therefore, the addition of GO content affected the character of membranes and pore density structure, which made an excellent opportunity to separate the impurities from wastewater especially for chrome metal rejection (Ajari et al., 2019). This type of studies previously stated for polypropylene and for low-density polyethylene (Othman et al., 2016; Ajari et al., 2019).

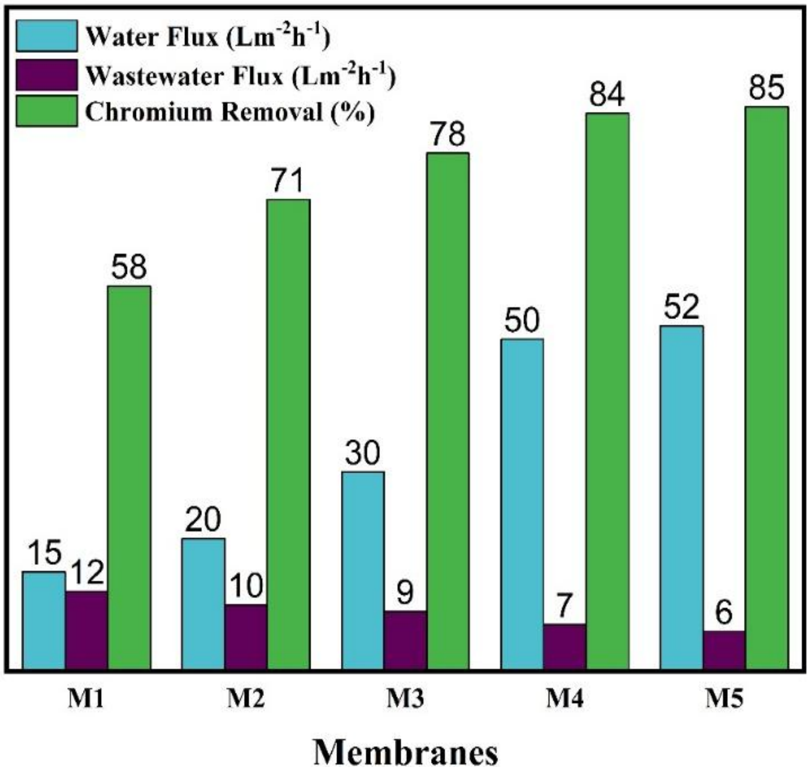

Figure 5: Performance of the developed membranes for wastewater treatments

4. CONCLUSIONS

In this research, using TIPS method HDPE/GO composites membranes were developed with incorporation of GO prepared by Hummers method resulting large amount hydrophilic groups with low cost mineral oil solvents. GO incorporation with HDPE has presented impacts on hydrophilicity, morphology, mechanical properties, permeability. For increasing hydrophilicity of developed membranes with doping of small amount of GO, water permeation and chromium rejection were improved. Hence, this membrane was effective for rejecting wastewater, with a chrome rejection of up to 85%. The 0.15% GO loaded HDPE/GO membrane exhibited 50 $Lm^{-2}h^{-1}$ water permeability and 84% chrome rejection during the filtration of wastewater but lower chrome flux yields which was occurred for denser pore size with increasing the incorporation of GO. As well, the mechanical properties was enhanced with incorporation of GO which is very practically importance in membrane technology for backwashing application. These membranes will be effective in removing contaminants especially chromium metal which has bad impacts on environment.

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