Energy Nexus 7 (2022) 100116

Contents lists available at ScienceDirect

Energy Nexus

journal homepage: www.elsevier.com/locate/nexus

An investigation into the efficiency of microalgal dynamic membrane photobioreactor in nickel removal from synthesized vegetable oil industry wastewater: A water energy nexus case study

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ARTICLE INFO

Keywords:
Wastewater
Nickel
Microalgae
Dynamic membrane photobioreactor
Vegetable oil
Heavy metals
Chlorella vulgaris

ABSTRACT

With growing concerns toward heavy metal pollution in wastewater due to their negative effects on human health and also the environment, a lot of effort has been put to find some novel and efficient methods in order to reduce or eliminate such hazardous elements. Chlorella vulgaris microalgae has been successful in heavy metal removal hence, the current study tries to develop an effective nickel removal technique using the combination of membrane separation along with microalgae dynamic membrane (DM) plus Chlorella vulgaris suspension in order to treat the synthetic vegetable oil wastewater. The experiments were divided into three phases. First phase was to comprehend the effect of microalgae's dry weight (DW) on nickel removal efficiency. With nickel's initial concentration being 10 mg. L^{-1}, the results indicated that by tripling the concentration of microalgae, the removal efficiency increases by more than 66% within the first hour of treatment. There was no significance increase in treatment efficiency by increasing the treatment time from 1 h to 24 h. In phase 2, by initializing the nickel concentration to 10,12.5,17.5 and 20 mg. L^{-1}, the experiments were done in a continuous mode inside a DM bioreactor (DMBR) after the microalgae DM was formed, which led to nickel's removal efficiency being reduced from 60% of previous phase to 22% after 1 h. In the third and last phase, fluidized microalgae inside a photobioreactor (PBR) plus micro-algae DM (DMPBR) were put in use. Comparing the results of this phase with last two phases, this phase with 72% removal compared to 63.6, 52 of previous phases, had the best results yet. To conclude, forming a dynamic membrane, not only preserves the primary membrane but also enhances the heavy metal removal efficiency.

1. Introduction

Vast quantities of organic and inorganic compounds are released into the environment annually because of human activities. Among these compounds are heavy metals, which are discharged from domestic plus industrial wastewaters into the water resources and will cause some drastic changes in those aquatic systems and their living organisms [1,2]. Heavy metals like nickel, cadmium, copper etc, have become the global concern in recent years because of their toxicity, accumulation and concentration in living organisms [1–3]. Also, at higher concentrations, heavy metal ions released from unspecific complex compounds in the cell, will lead to toxic effects [4,5].

Nickel is used in many industries and large amounts of nickel can be found in their effluents. The recommended standard for Ni in industrial effluents by WHO is 0.02 mg. L^{-1}, while US-EPA suggested 0.01 mg. L^{-1} for this element. The average concentration of Ni in Netherland's ground water ranges from 7.9 µg. L^{-1} (in urban areas) up to 16.6 µg. L^{-1} (in rural areas) [2,6].

The World Health Organization (WHO) has declared that the amount of nickel metal ions in human-consumable water should not exceed 0.02 g. L^{-1}. High concentration of nickel in aqueous solutions may cause severe damages to human health, which include, but are not limited to, damages to lungs and kidneys, skin dermatitis, and renal edema [7–9].

Certain food items contain high percentages of nickel, such as cocoa (up to 8.2 -12 mg.kg^{-1} fresh wet weight), dark chocolate, soya beans, oatmeal, nuts, and almonds, to name a few. From this, it can be concluded that industries using such items may have some amount of nickel in their wastewater [10].

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https://doi.org/10.1016/j.nexus.2022.100116
Received 24 November 2021; Received in revised form 4 July 2022; Accepted 6 July 2022
Available online 15 July 2022

Table 1
Common heavy metal treatments systems.

Treatment system	Heavy metal concentration ($mg.L^{-1}$)	Heavy metal removal efficiency (%)	Refs.
Ion exchange	200	Co (III): 100Ni (II): 100Cr (III): 100	[15]
Reverse osmosis	50 to 200	Cu(II): 96Pb(II): 97.5Ni(II): 98.5	[24]
Electrochemical treatment	Cu: 3810Ni: 3520	Cu: 98Ni: 45	[25]
Electrodialysis	22.4 and 24.4	Cu (II): >99 Ni (II): >99	[26]

Table 2
Some common biological systems for heavy metal removal.

Treatment system	Heavy metal concentration (mg. L^{-1})	Heavy metal removal efficiency (%)	Refs.
Activated sludge	10 to 100	at 100 mg. L^{-1}metal concentration: Cu (II): 100 Cr (III): 85 Co (II): 80 Zn(II): 100 Cd(II): 90 Ni(II): 25 Cu(II): 96 Pb(II): 97.5	[28]
MBR+eggshell Diatoms	Al (III): 6 Fe (II): 6.5 Zn (II): 12 As (III): 5.27 Ag(I): 4.28 Ni(II): 3.95 Cr(VI): 4.09 Pb (II): 4.081	Al (III): 97 Fe (II): 74 Zn (II): 59 As (III): 96.67 / 96.48 Ag(I): 98.52 / 98.46 Ni(II): 95.24 / 95.44 Cr(VI): 7.33 / 9.29 Pb (II): 98.82 / 98.80	[29] [30]
Biochar	200 mg. L^{-1}	Ni (II): 45 to 87	[31]

To add to the above-mentioned cases in the food industries, edible vegetable oils undergo a hydrogenation process, which is one of the most important parts of edible oil processing in order to achieve products with predetermined physical properties and chemical stability beyond the liquid form in the earlier phase, and hydrogenation needs nickel as a catalyst [11,12]. It has been declared that the amount of nickel used annually is 500,000 to 1,000,000 pounds for making 2.5 billion pounds of vegetable oil [13].

Several methods have been developed in order to remove nickel over the years which have been commonly used, methods such as chemical precipitation [14], Ion exchange [15], activated carbon adsorbents [16,17], electro dialysis [18], electrochemical treatment [19] and reverse osmosis [20]. However, these techniques have many drawbacks such as having high operating costs, being dangerous to the environment and energy consuming [21–23]. Table 1 represents some of the most common wastewater treatment systems used for heavy metal removal.

On the other hand, many living microorganisms (e.g., algae, fungi, bacteria and yeast) have been widely studied because of low operating cost, and being ecofriendly, metal bio sorption from polluted waters are becoming more popular. Among them, micro algae have proved to have the highest metal bio adsorption capacities, this is because of their cell walls, which are made of a fiber-like structure, plus from their shapeless embedding matrix of various polysaccharides [27].

Table 2 shows some of the most common biological methods for heavy metal removal.

Plus, heavy metal removal by algal biomass is not only comparable with but also sometimes even higher than that of chemical sorbents [32].

It is known that biological nutrient removal is one of the most efficient ways for wastewater treatment [33–35].

Microalgae are photosynthetic microorganisms which wield energy from the sun or artificial light sources to grow and consume inorganic nutrients and CO_2 [36,37].

Wastewater treatment using microalgae, can offer ecologically safe, relatively inexpensive, and more effective ways to remove nutrients and metals from wastewater than the conventional methods [38]. Also, Hatamifard et al. have proved that algae are more effective in heavy metal removal than the activated sludge [39].

Table 3 is a brief summary of several microalgal treatment systems contaminated by wastewaters containing heavy metals.

Compared to most water treatment technologies, membranes have a competitive advantage in treating alternative nutrient streams due to simplicity of automation, relatively small footprint required, and low sensitivity to many influent water quality parameters (e.g., pH, temperature, dissolved nutrients) [44–46]. Submerged membrane technology has been chosen due to a lower energetic consumption compared to tangential filtration. Submerged membranes are already used in membrane bioreactor (MBR) for wastewater treatment [47,48].

As combination of membrane separation and wastewater treatment using microalgae goes, membrane photobioreactor (MPBR) has shown a good performance both in microalgae biomass production and nutrients removal [39]. In addition, the generated algal biomass, can be used to produce biofuel [36], lipid and protein production [49], CO_2 capturing [50] etc. Therefore, it can be deduced that MPBR is a competitive technology for the treatment of wastewater [51]. Plus, MPBRs have shown to provide a high controllability on microalgae retention inside the operation system because of a strict regulation in solid and hydraulic retention time (HRT and SRT) [52].

However, the main disadvantage of membrane separation is a phenomenon called membrane fouling, which is due to the algal cake layer formed on the membrane's surface, which can lead to an increase in energy demand as flow decreases and hydraulic resistance increases [53,54]. However, this issue can be taken advantage of by forming a secondary algal membrane as a DM on the static membrane in order to increase the separation efficiency [55]. In this system, in addition to biological nutrient degradation, physical separation will take place simultaneously with the help of DM, as microalgae have been shown to be very good at removing heavy metals from wastewater. Other benefits of DM include improving separation efficiency and reducing the high cost of membrane recovery [39]. It is worth noting that DM can be easily

Table 3
A brief summary of the methods for heavy metals removal using microalgae.

Microalgae species	Treatment system	Heavy metal concentration (mg. L^{-1})	Heavy metal removal efficiency (%)	Refs.
Scenedesmus acutus / chlorella vulgaris Scenedesmus acutus / Chlorella vulgaris	Kappa-carrageenan (Fluidized bed) Polyurethane foam (Packed bed)	Cd: 5 Zn: 300 Cr: 1	Cd: 73 / 66 Zn: 91 / 85 Cr: 36 / 48 Cd: 69 / 57 Zn: 84 / 78 Cr: 31 / 34	[40]
Chlorella vulgaris	Erlenmeyer (batch system)	nNiO: 10-50	nNiO to nNi bio-reduction: 85	[41]
Chlorella vulgaris	Dynamic membrane photo bioreactor	Hg: 0.4-0.8	Hg: 78.16	[39]
Fucus vesiculosus	Continuous system using A-PEI material	25-30	Cd(II): 56.22 Ni(II): 47.85 Pb(II): 74.76 Cu (II): 55.60	[42]
Chlorella vulgaris	Membrane photo bioreactor	Cr: 0.5-5	Cr: 41.9, 50	[43]

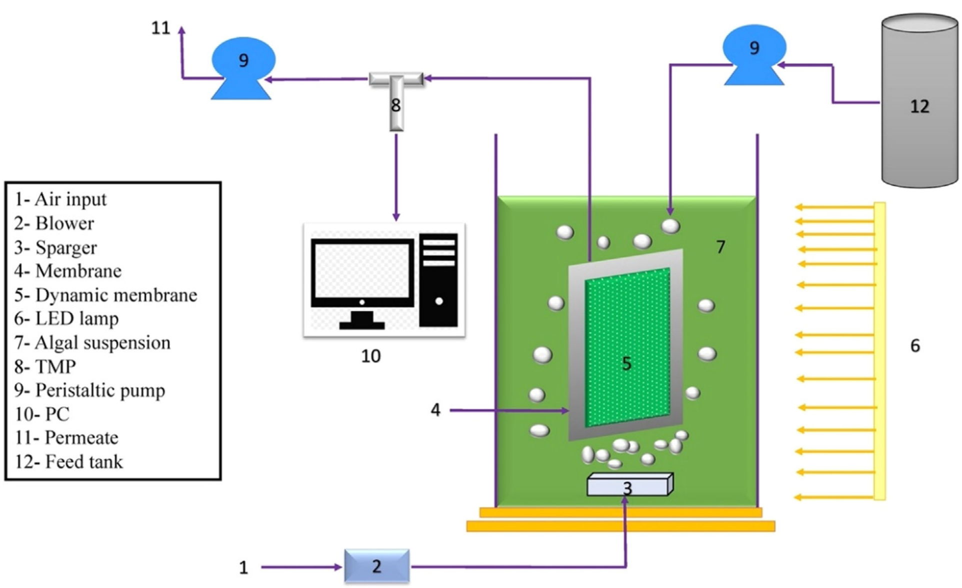

Fig. 1. Schematic diagram of dynamic MBPR.

removed from the membrane, either by simple washing or by washing in the reverse flow of air or water [56].

Some investigations have been made on heavy metal removal using algal suspension but, as no investigation has been made on nickel removal using both algae DM and algal suspension inside a DMPBR, it became the purpose of this study to investigate and compare nickel's removal efficiency from synthetic vegetable oil wastewater using this technique in three different phases including removal efficiency using microalgae suspension, microalgae DMBR and DMPBR.

2. Materials and method

2.1. DMPBR setup

The DMPBR was made of plexiglass with the height of 60 cm, length of 15 cm, width of 10 cm, and the working volume of 4 liters which is schematically illustrated in Fig. 1. A sparger was installed at the bottom of the DMPBR, which was connected to an air pump with aeration rate of 4.5 lit.min^{-1}. A polymer membrane was used in the experiment as the static membrane with pore size of approximately 0.4 μm and effective surface area of 0.048 m ^{2}. The membrane's distance from the sparger was 5 cm. A computer and a Trans-Membrane Pressure (TMP) gauge were employed. Fig. 2 represents the formation of a microalgae-dynamic membrane on a static membrane.

2.2. Microalgae and nickel suspension preparation

The microalgae used in this experiment were *Chlorella vulgaris*, which is a kind of green algae well known for its heavy metal adsorption and round shape. For microalgae cultivation, BG 11 culture medium was used [57]. 5% of microalgae were inoculated to the 10-liter bubble column photobioreactor and transferred to the MPBR after the biomass growth. All experiments were performed under laboratory conditions at 25°C. Also, the microalgae light/dark regime was 24:0 under 27 μmol.m^{-2}s^{-1} white LED illuminating the MPBR. Moreover, the initial DW of the microalgae was set to 0.206 g. L^{-1}.

As nickel removal has been the main objective of this study, and vegetable oil wastewater has many other components besides nickel, it was decided to synthesize one instead of using the real one. The nickel was obtained from Nickel nitrate hexahydrate salt with the chemical formula of Ni $(NO_2)_3$ .6H NiO and molar mass of 290.81 mol. g^{-1} (Merck – Germany) Nickel concentrations were 10, 12.5, 17.5 and 20 mg. L^{-1} which was similar to that in Kant Mehta's study [58].

To investigate the most efficient method in this study, three different phases were designed, first phase was about investigating nickel removal

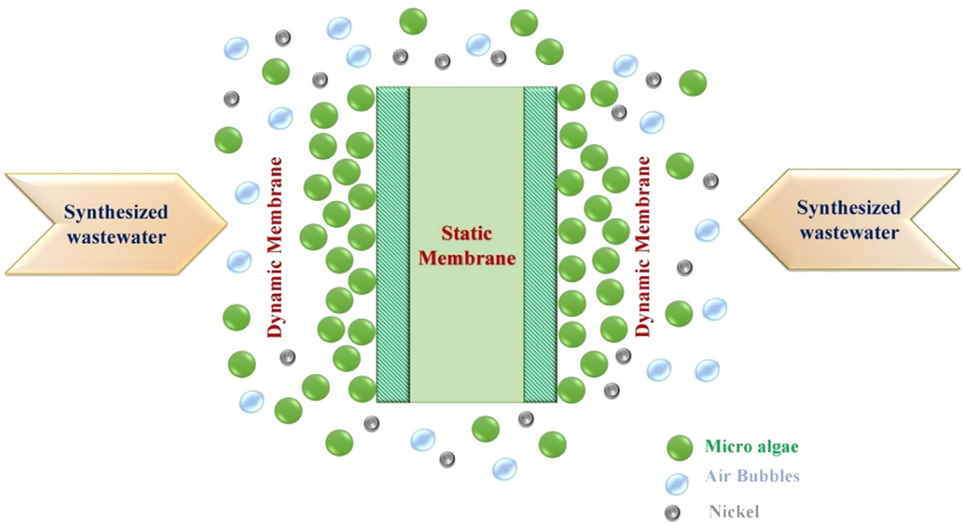

Fig. 2. formation of microalgae-dynamic membrane.

Table 4
A summary of taken steps.

Row	Phase	Description
1	Suspension (phase 1)	Investigating nickel removal using microalgae suspension within an Erlenmeyer flask
2	DMPBR (phase 2)	Investigating nickel removal using algal dynamic membrane within the membrane photo bioreactor
3	DMPBR and suspension (phase 3)	Investigating nickel removal using algal dynamic membrane and microalgae suspension within the membrane photo bioreactor simultaneously

using microalgae suspension, phase two put algae DM in use for this purpose and lastly phase three was the combination of these two phases. Table 4 represents a summary of the mentioned steps.

In phase 1, Four Erlenmeyer flasks each with volume of 250 ml were used to observe the removal of nickel using micro algae suspension.

As for phase 2 a DMBR was used to witness the same procedure as in phase 1 but with microalgae DM. Firstly, a micro-algae dynamic was formed elsewhere and then, it was put inside the photobioreactor. This is done by measuring Trans Membrane Pressure (TMP). As algae-dynamic membrane forms, TMP steadily rises which shows the quantity of microalgae on the membrane's surface. It was concluded that if TMP is in the range of 300 mbar then algae DM has been formed [39].

In phase 3, just as phase 2, soon after the microalgae DM's formation was completed, it was placed within the DMPBR however, for this phase the biological treatment was not only up to microalgae DM, but also to the suspension of the living microalgae.

The removed nickel was analyzed by atomic adsorption test using Varian's SpectrAA-200 model.

3. Results and discussion

3.1. The effect of microalgae suspension on nickel removal

In this phase of experiments, an investigation of the effect of microalgae suspension on nickel removal efficiency was made. Two times were assumed (1 h and 24 h). Moreover, a constant concentration of nickel (10 mg. L^{-1}) and two DWs of microalgae (0.062, 0.206 g. L^{-1}) were considered in order to find the most efficient time and DW for nickel removal, this is shown in Fig. 3.

As it is comprehendible from the results, there is a significant difference between the results, for DW = 0.062 g. L^{-1} a 36% of nickel removal efficiency was reached and for the DW = 0.206 g. L^{-1} a 60% of nickel removal efficiency at first hour of microalgal treatment was reached. In other words, by tripling the concentration of microalgae nickel removal efficiency increases by more than 66% in the first hour of the treatment. Also, there is no significant increase in treatment efficiency by increasing the treatment time from 1 h to 24 h and the greatest amount of heavy metal removal occurs in the first hour of the biological treatment process meaning that the rate of removal in the next hours of the process can be ignored. This result is regarded in Pahlevanzadeh et al. report [59].

3.2. The effect of microalgae DM on nickel removal

In the second phase of the experiments, after the DM had been formed, it had to begin the treatment of 4 liters of vegetable oil synthetic wastewater in the DMBR and after 30 and 60 min, the samples were collected. This time, nickel concentrations were between the initial value of 10 mg. L^{-1} to 12.5 mg. L^{-1}, reaching 17.5 mg. L^{-1}, and 20 mg. L^{-1}. The samples were collected from the permeate stream from DM. Fig. 4 represents the results for microalgae DMBR efficiency in nickel removal.

Comparing the first and second phase, at DW= 0.206 g. L^{-1}, nickel concentration was 10 g.L^{-1}, it is clear that nickel removal efficiency is reduced from 60% in phase 1 to 22% in phase 2. compared to phase 1, the nickel removal efficiency has decreased more than 63%. Since in phase 1, biological nickel removal has been occurred by *Chlorella vulgaris* suspension in 250 ml Erlenmeyer in batch mode. On the other hand, in phase 2, nickel was eliminated in continuous mode in 4 Liters DMBR in the absence of microalgae suspension resulting in nickel separation only by microalgae DM from vegetable oil synthetic wastewater.

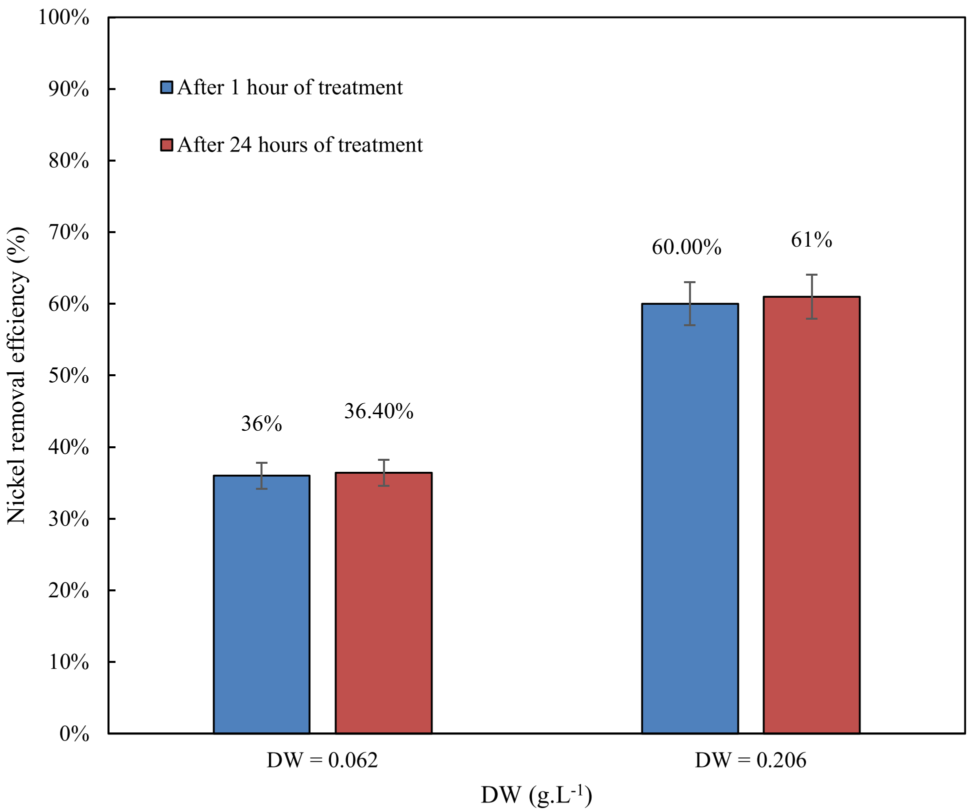

Fig. 3. Nickel removal efficiency vs. DW.

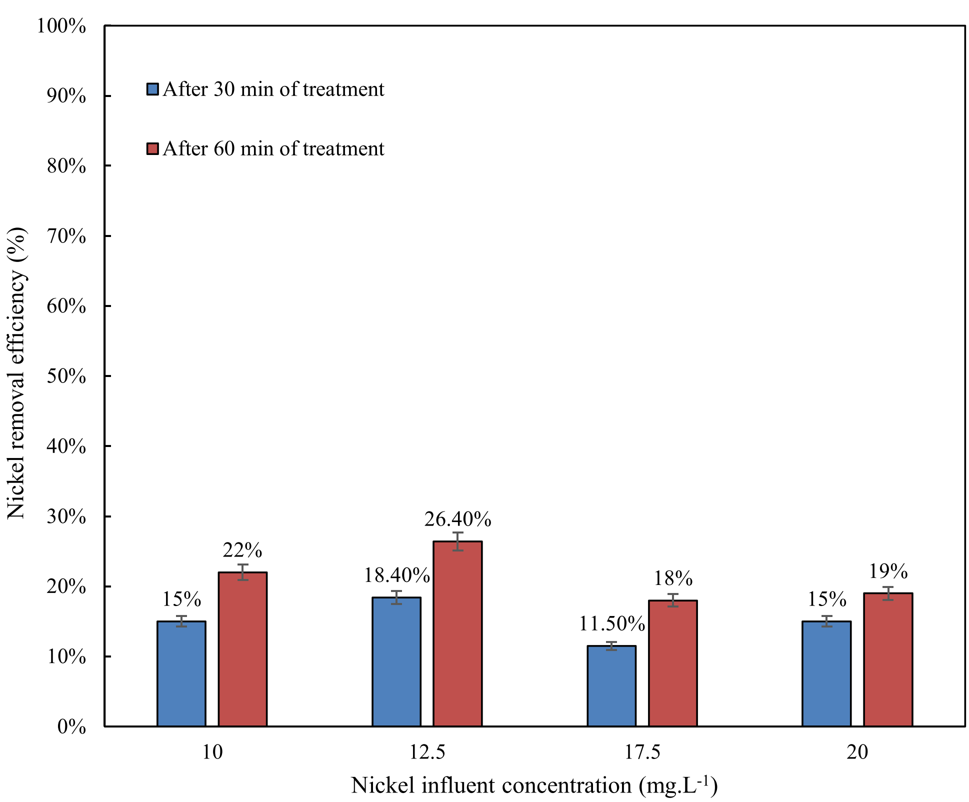

Fig. 4. Microalgae DMBR nickel removal efficiency vs. nickel influent concentration.

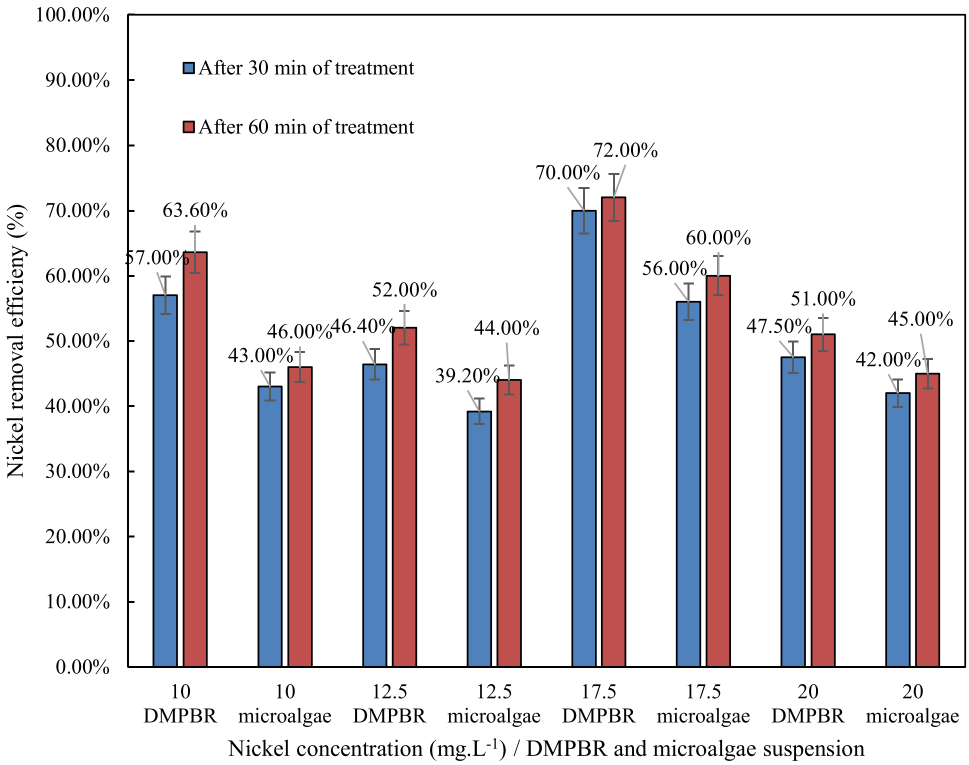

Fig. 5. Nickel removal efficiency vs. using microalgae DMPBR and microalgae suspension and nickel influent concentration.

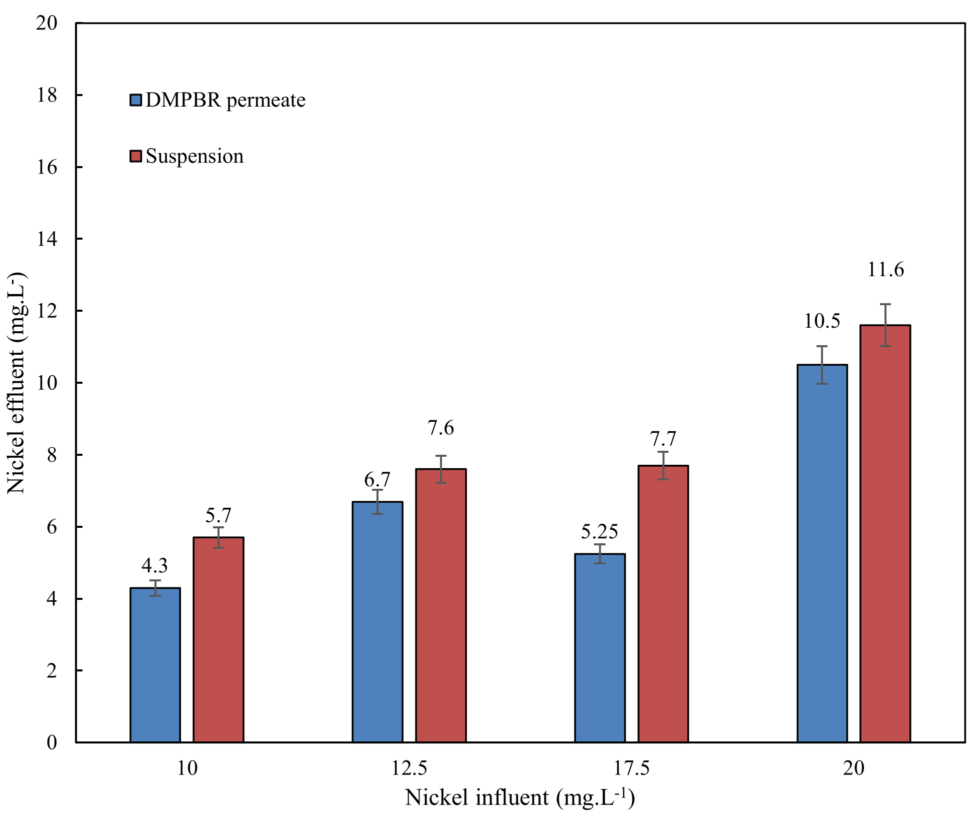

Fig. 6. Nickel effluent vs. nickel influent concentration after 30 min of treatment in DMPBR.

It is a common fact that Fluidized (suspended) sorbents not only provide a superb mixing, but also maintain the phases fully mixed at all times in addition to all the mentioned items, mass transfer rate is improved among the phases [60–63]. As in this condition, sorbents are in a constant movement through the photo bioreactor, moving and migrating within it thoroughly and contacting the pollutants in high rate [62,64].

On the other hand, there is immobilization or fixing the sorbents methods. Such methods are done in a way that will limit the practical's movement of atoms, molecules, and substances of biological material

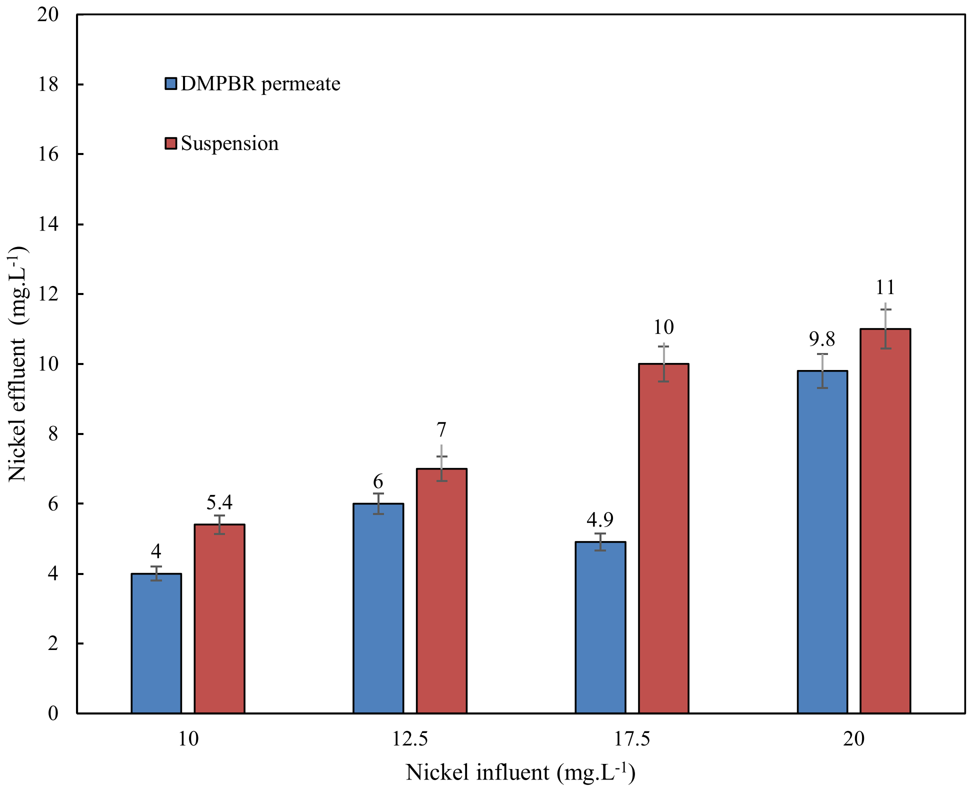

Fig. 7. Nickel effluent vs. nickel influent concentration after 60 min of treatment in DMPBR.

Table 5
A comparison of nickel effluent between phase 2 and phase 3.

Treatment phase	Ni influent (mg. L^{-1})	Ni effluent in microalgae suspension(mg. L^{-1})30 / 60 min	Ni effluent in membrane permeate(mg. L^{-1})30 / 60 min	Microalgae DMBR efficiency (%)30 / 60 min	Chlorella vulgaris suspension efficiency (%)30 / 60 min	Microalgae DMBPR efficiency (%)30 / 60min
2	10	-	8.5 / 7.8	15 / 22	-	-
	12.5	-	10.2 / 9.2	18.4 / 26.4	-	-
	17.5	-	15.48 / 14.76	11.5 / 18	-	-
	20	-	17 / 16.2	15 / 19	-	-
3	10	5.7 / 5.4	4.3 / 3.64	14 / 14	43 / 46	57 / 63.6
	12.5	7.6 / 7	6.7 / 6	7.2 / 8	39.2 / 44	46.4 / 52
	17.5	7.7 / 10	5.25 / 4.9	14 / 12	56 / 60	70 / 72
	20	11.6 / 11	10.5 / 9.8	5.5 / 6	42 / 45	47.5 / 51

partially or entirely on a solid base or within some unique constructions. Comparing fixed methods to fluidized methods, there is a much higher risk of clogging and also lower adsorption efficiency since fluidization maximizes the surface contact between bio-sorbents and pollutants [60,65].

Same goes for this experiment. Meaning that, this is the reason behind more nickel removal efficiency in the first phase than the second. It can be argued that mobilized bio-sorbents offer much higher absorption efficiency than immobilized microalgae on the surface of membrane (dynamic membrane). As microalgae is able to move freely throughout the photo bioreactor, there is much higher chance of contacting between nickel and microalgae plus there is no clogging occurrence.

This result is essential as it shows the position of the next phase. For the next phase all the considerations will not be changed except one, in the following stage a microalgae DM along with algal suspension will face the same concentrations of nickel. It is anticipated that in the next phase, more nickel would be removed.

3.3. The effect of combination of microalgae DM and microalgae suspension in DMPBR system on nickel removal

This peculiar phase was probably the most complex and the hardest one in which by the same PBR, the maximum removal percentage was targeted. This phase was in fact a combination of the first and the second phases. After forming the DM, it was put inside the DMPBR. However, nickel contaminated wastewater was added to the algae suspension. This was because, as observed in the first phase, *Chlorella vulgaris* itself had the potential to remove nickel. Hence, combining microalgae DM and microalgae suspension could be a very promising capability. Fig. 5 illustrates the results for this phase. The concentration of microalgae in this phase was similar to previous phases (DW = 0.206 g.L^{-1}), since the objective of this phase was to determine the removal efficiency of algal dynamic membrane along with microalgae suspension. It is obvious that by changing the microalgae's concentration, whether suspension's or DMPBR's, the yielded results would be untrustworthy.

As expected, combining the first two phases provided the best results. Reaching the maximum level of nickel removal, at 72%, was quite satisfying. Like in the previous experiment, we observed that there was a significant difference between the 30 min and 60 min samples, whether they were obtained from microalgae DM or algae suspension.

Notice how much higher nickel removal percentage was achieved by using DMPBR. It is an astonishing result, proving that the method is totally effective. The final outcome is that using both microalgae DM and microalgae suspension can be the most effective method of removing nickel from industrial wastewater.

Figs. 6 and 7 illustrate the nickel influent and effluent after 30 and 60 min treatment in DMPBR, respectively. The illustrations vividly show that in phase 3, microalgae DMPBR had the best nickel removal results compared with two previous phases.

Table 5 represents a comparison of nickel effluent between phase 2 and phase 3. This chart shows that in phase 3 much more nickel has been removed. This is because of the existence of micro algae's suspension. After three times of repetition, the average difference between the effluents in 30 min is 6.10 mg. L^{-1} and in 60 min is 5.81 mg. L^{-1}, which shows that in 30 min of treatment time more nickel will be treated than in 60 min of treatment time.

4. Conclusion

Concerning heavy metal removal, MPBRs have shown to be an excellent method for this purpose. Using Chlorella micro algae's suspension along with its formed DM proved to be the most fruitful combination in nickel elimination reaching 72% of its removal within an hour, while using suspended microalgae or algae-dynamic membrane led to maximum 60% and 22% of nickel removal respectively. Managing the water-energy nexus has become a major challenge in many parts of the world, especially in the Middle East. Due to limited water and energy resources and climate change, it is time to consider water as a reusable resource rather than a consumable one. In addition, the energy consumption of wastewater systems should also be considered, as many of them consume a large amount of energy to treat wastewater, which is not a logical trade-off between water and energy. This has led scientists to come up with an effective, and green method to treat wastewater. This novel algal dynamic MPBR, being eco-friendly, and low energy demanding is an ideal technique for commercial practice in nickel elimination from wastewater.

Acknowledgements

We would like to thank Ms. Mozhdeh Karbalaee Hosseini Ghiasvand for drawing the figures with care

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