Preparation of a Cellulose Filter from Palm Fronds used for Treating Industrial and Domestic Polluted Wastewater

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Abstract:
This study was conducted in the laboratory of the Faculty of Biology/Pure Science Education/Anbar University to investigate the efficiency of palm leaves as plant waste for purification of domestic and industrial wastewater. Tests were conducted before and after treatment of wastewater using cellulose membranes made of cellulose acetate from palm leaves. The results showed that pH values were balanced and optimum turbidity removal of 82% and 86% for domestic and industrial wastewaters was achieved respectively. Good results were achieved for sodium and potassium removal, with 81% sodium removal for domestic wastewater and 78% for industrial wastewater. Potassium removal was 82% for domestic wastewater and 74% for industrial wastewater. Total dissolved solids (TDS) test results showed a reduction of 25% and 20% for domestic and industrial wastewater respectively. Chloride removal was 35% for domestic wastewater. Results for nitrates showed a removal of 22% for domestic wastewater and 25% for industrial wastewater. Removal of phosphate, sulphate and total hardness was 41%, 25% and 33% for domestic wastewater respectively. Alkalinity showed balance within permissible limits. For total suspended solids, the cellulose filter had a high removal efficiency of 93% for domestic wastewater and 91% for industrial wastewater. The BOD was reduced from 25 mg/L to 13 mg/L for domestic wastewater and from 18 mg/L to 13 mg/L for industrial wastewater. In the Total Colony Count (TPC) test, the removal efficiency was 97% for domestic wastewater and 96% for industrial wastewater.

Keywords: Palm fronds, Cellulose filter, Industrial wastewater, Domestic wastewater

تحضير فلتر سليلوزي مستخرج من سعف النخيل واستخدامه في معالجة المياه العادمة الملوثة الصناعية والمنزلية

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المستخلص:
اجريت الدراسة الحالية في مختبرات قسم علوم الحياة/كلية التربية للعلوم الصرفة/جامعة الانبار لاختبار كفاءة سعف النخيل كمخلفات نباتية في تنقية المياه العادمة المنزلية و الصناعية، اجري فحص للمياه العادمة قبل اجراء المعالجة عليها وبعدها تم استخلاص السليلوز على شكل خلات السليلوز من سعف النخيل وتحويله الى غشاء سليلوزي.

بينت النتائج التي تم الحصول عليها ان ال pH في حالة توازن وان افضل نسب ازالة للعكور بلغت 82% للمياه العادمة المنزلية بينما للمياه العادمة الصناعية بلغت 86% وكانت الازالة فعالة فيما يخص الصوديوم والبوتاسيوم اذ كانت النسب المئوية للإزالة للصوديوم للمياه العادمة المنزلية 81% وللمياه العادمة الصناعية 78% ، اما النسبة المئوية للإزالة للبوتاسيوم بلغت للمياه العادمة المنزلية 82% و للمياه العادمة الصناعية 74% اما فيما يخص فحص المواد الصلبة الذائبة الكلية فبينت النتائج انخفاض بنسبة 25% و20% للمياه العادمة المنزلية والصناعية على التوالي ، اما الكلوريدات كانت النسبة المئوية للإزالة 35% للمياه العادمة المنزلية اما النترات فكانت النتائج تشير الى ان نسب الازالة وصلت الى 22% للمياه العادمة المنزلية و 25% وللمياه العادمة الصناعية ، فيما سجلت كل من الفوسفات، الكبريتات ، العسرة الكلية نسب مئوية للإزالة بلغت 41% ، 25% ، 33% للمياه العادمة المنزلية على التوالي واظهرت القلوية توازن ضمن الحد المسموح به اما فحص المواد الصلبة الكلية العالقة فقد حقق المرشح السليلوزي كفاءة عالية في الازالة اذ بلغت النسبة 93% للمياه العادمة المنزلية و91% للمياه العادمة الصناعية ، فيما انخفض المتطلب الحيوي للأوكسجين من 25 الى 13 ملغم /لتر للمياه العادمة المنزلية ومن 18 الى 13 ملغم /لتر للمياه العادمة الصناعية اما فحص العدد الكلي للبكتريا TPC فكانت النسبة المئوية للإزالة 97% و96% للمياه العادمة المنزلية والصناعية على التوالي.

الكلمات المفتاحية: سعف النخيل، فلتر سليلوزي، مياه عادمة منزلية، مياه عادمة صناعية

Introduction

Rapid population growth and widespread environmental pollution have exacerbated water shortages caused by poor management [1]. Water pollutants such as pharmaceuticals, minerals, dyes, metals, pesticides, fertilizers, and other sources of pollution, whether domestic, industrial, or agricultural, are a major environmental problem [2]. All of these pollutants enter wastewater systems and threaten all living organisms [3]. As a result, a variety of water purification methods have been developed. Considering that the most cost-effective and efficient methods are optimal and in demand in water purification processes [4], membrane water

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treatment has emerged as a promising and cost-effective technology to expand water resources and address water pollution issues. It is expected to play a more important role in water purification and water reuse, such as domestic sewage [5].

A membrane is a selective barrier between two adjacent phases that regulates the transport of materials [6]. The porosity, permeability, and selectivity of a membrane depend on the material used in its construction [7]. In general, membrane materials must be thermally and chemically stable, have high mechanical strength, and be able to form flat membranes or porous fibers [8]. Many industrial membranes have demonstrated their ability to separate pollutants from water [9]. This technology is widely used in water treatment and continues to attract increasing attention due to its high efficiency in removing particles, turbidity, and microorganisms from wastewater [10]. Membrane technologies are becoming increasingly important as they can effectively and reliably remove pollutants without forming harmful byproducts, especially in wastewater treatment and removal of heavy metal ions [11].

Cellulose is one of the materials used to make filtration membranes. Cellulose filters are an excellent and cost-effective choice [12]. Cellulose has the advantages of high availability, low cost, easy access, non-toxicity, and biodegradability. Their membranes are particularly suitable for removing small concentrations of pollutant particles from water. [13].

Research objectives

1. Extracting cellulose from palm fronds
2. Converting cellulose into a cellulose membrane to treat industrial and domestic polluted wastewater.

Materials and Methods

Extraction of cellulose from palm frond waste

Physical treatment

Palm fronds were collected, cleaned, and cut into small pieces. Next, they were dried at 60°C in an oven for 48 hours. Then, they were ground to obtain a fine powder to complete the remaining extraction stages [14].

Chemical treatment

Chemical treatment was carried out on palm fronds to extract cellulose following the steps below:

1- Removing pectin and water-soluble substances

A sample of 30 g of palm fronds was soaked in deionized water at a ratio of 1-80 g/L with continuous stirring at 50 °C for 2 hours. Then, the sample was filtered and dried in an electric dryer at 60 °C [15].

2- Removing lipid substances

Lipids were removed using a Soxhlet extractor, where toluene was mixed with ethanol in a 2:1 ratio. The Soxhlet extractor was prepared at 180°C, and 500 ml of the mixture was placed in the spherical flask and left for 7 hours. After the time had elapsed, the sample was washed with 30% diluted ethanol, filtered and washed with deionized water, and placed in the oven at 60°C to dry until completely dry [16].

3- Treating with sodium hydroxide

This treatment aimed to remove hemicellulose by immersing the sample in a 20% sodium hydroxide (NaOH) solution, with a ratio of one part sample to two parts solutions, for 1.5 hours at 170°C. After the specified time had elapsed, the mixture was filtered and washed with deionized water until the pH of the washing water became neutral. Finally, the sample was left to dry in an oven at 60°C until completely dry [17].

4- Bleaching stage

It is a chemical process to eliminate the remaining lignin, obtain pure cellulose, and bleach the sample's color to white. This process is performed with sodium chlorite $NaClO_2$ at a concentration of 1% and a pH of 5 and at a ratio of one volume of the sample to two volumes of the solution for 60 minutes and a temperature of 70°C. After the specified time had elapsed, the sample was filtered and washed several times until reaching a pH of 7 [18].

5- Obtaining cellulose acetate

At this stage, 1g of the cellulose obtained was added to a mixture of 10 ml of glacial acetic acid (CH_3COOH) and 1 ml of sulfuric acid (H_2SO_4). The mixture was heated to 80°C for half an hour until the cellulose dissolved. After cooling, 10 ml of acetic anhydride ($C_4H_6O_3$) was added, and the sample was heated to 70°C for 15 minutes. When the specific time was elapsed, 400 ml of deionized water was added to stop the reaction. The precipitate was filtered and washed with deionized water until reaching a pH of 7 and dried in an oven at 60°C [19].

6- Preparing cellulose membrane from cellulose acetate

Once cellulose acetate was obtained, it was ground into a fine powder. Next, 0.5 g of cellulose acetate was dissolved in 10 ml of chloroform and then poured onto a fine sieve mesh to provide support for the membrane during the process of filtering polluted water [20].

Physicochemical and biological tests

The physicochemical and biological tests below were performed on the samples before and after treatment, as follows:

1-pH: The pH was measured using a pH meter, according to the method of [21]

2-Turbidity:

Turbidity was measured according to procedure [22] using a Turbidity meter.

3-Sodium (Na) and Potassium (K):

The sodium and potassium values were calculated with a Flame photometer.

4-Total Dissolved Solids (TDS):

The total dissolved solids concentration was tested relying on the procedure [21].

5-Water Electrical Conductivity (E.C): Electrical conductivity was measured with a PH-EC-TDS meter equipped by Hanna HI-9812 Company.

6-Chloride (Cl-):

Chlorides were measured using the titration method only [22].

7-Nitrates (NO_3):

Nitrates were measured by a multiparameter photometer (Hl 83200).

8-Phosphate (PO_4):

It was measured by a multiparameter photometer (Hl 83200).

9-Total Hardness (TH):

It was measured using the titration method [22].

10-Sulfate (SO_4):

It was measured according to the method adopted by [21].

11-Total Alkalinity (TA):

The titration method was used to measure total alkalinity according to [21].

12-Total suspended solids (TSS):

It was calculated according to the method [21].

13-Biochemical oxygen demand (BOD):

The biological requirement was measured with a Dissolved Oxygen meter (HI98193) indirectly, where the amount of free dissolved oxygen (DO) was measured using a water quality meter before and after incubation in the dark for five days at 30 °C to prevent photosynthesis (and thus the addition of oxygen). The dissolved oxygen level was measured again and calculated according to APHA (2017) and expressed by mg/L unit, following the equation BOD5 = DO1-DO2.

14-Total Plate Count (TPC):

It was calculated using the pouring dishes method, as described in the procedure [23].

Table 1. The physicochemical and biological tests of wastewater before treatment with plant waste

Test	Measure unit	Domestic wastewater	Industrial wastewater
pH		6.9	6.7
Turbidity	NUT	75	150
Na	mg\L	400	301
K	mg\L	33	31
TDS	Ppm	1202	1105
E.C	µS/cm	2100	2000
Cl-	mg\L	234	170
NO_3	mg\L	3.2	2.8
PO_4	mg\L	0.17	0.12
TH	mg\L	1500	375
SO_4	mg\L	400	250
TA	mg\L	125	166
TSS	mg\L	511	442
BOD	mg\L	25	18
TPC	Cell/ml	960	840

Results and Discussion

In this study, cellulose membranes made of palm leaves were used after pouring cellulose acetate into a sieve. Domestic and industrial wastewater were filtered at a rate of 350 ml/min at a pressure of 1.5 bar. The results in Table 2 and Table 3 show that the pH values of both waters, domestic and industrial wastewater, were balanced, indicating a balanced water content [24]. The results also showed that turbidity removal was achieved at 82% for domestic wastewater and 86% for industrial wastewater, as the substances causing water turbidity either adhered to the membrane surface or were absorbed into the membrane pores, depending on the size of the membrane pores. The relationship between the particle size and the membrane pore size [25]. These results are consistent with those obtained in [26]. The results of the sodium (Na) and potassium (K) tests were promising. The removal efficiencies of sodium and potassium were 81% and 82% for domestic wastewater, respectively, and 78% and 74% for industrial wastewater, respectively. The high removal rates for sodium and potassium may be due to the fact that these ions can ionize and react with functional groups in the membrane, such as the hydroxyl groups (OH-) in the cellulose, to form potassium hydroxide (KOH) and sodium hydroxide (NaOH). These ions can also be removed by physical absorption, with some of them being retained [27]. These results are consistent with those of [28] and close to those of [29].

The results related to the total dissolved solids (TDS) measurement showed a 25% reduction for domestic wastewater and a 20% reduction for industrial wastewater, depending on the size of the solutes attached to the membrane, some of which can react with the hydroxyl groups (-OH) in the cellulose structure that make up the membrane [30]. The results for industrial wastewater are close to those of [31]. E.C. electrical connection tests showed a 43% reduction for domestic wastewater and a 49% reduction for industrial wastewater, depending on the dissolved salts that give the water this property [32]. In the chloride (Cl) test, the removal efficiency was 35% for domestic wastewater and 30% for industrial wastewater, because the principle of chloride removal by the membrane is absorption by binding to cellulose molecules or by ion exchange through interaction with sulfonic acid groups [33]. The results are lower than those obtained in [34] because the membrane used is modified and composite, rather than pure and simple. The results for nitrate (NO3) showed a removal efficiency of 22% for domestic wastewater and 25% for industrial wastewater. Since this relies only on physical absorption, the results are considered satisfactory. Previous studies have shown that modification of the membrane by adding materials such as amines can improve the removal efficiency [35]. In the phosphate test (PO4), the removal efficiency was 41% for domestic wastewater and 16% for industrial wastewater, because phosphate molecules are directly attached to the membrane surface and can also undergo ion exchange with the hydroxyl ions of the membrane [36]. The results are almost similar to those in [37].

When measuring total hardness (TH), the results showed a 32% removal for domestic wastewater, while the removal for industrial wastewater was lower at 21%. What is important here is the size of the particles, the capacity of the pores and the structure of the membrane that captures them [38]. The results for domestic wastewater are consistent with those of [39]. In the sulfate test (SO4), the percentage reached 25% for domestic wastewater and 20% for industrial wastewater. The removal process is affected by the ability to filter and retain sulfate molecules. It also depends on electricity, since sulfate has a negative charge and the membrane has a positive charge that attracts the molecules to the membrane [40]. The results are close to those of [41]. The total alkalinity (TA) results are within the permissible limits and are acceptable, since they are affected by the pH value, which represents the ability of water to accept protons, resulting in neutralization of water acidity [42]. As for the TSS test of total suspended solids, it depends on the filtration of water and the retention of suspended solid particles (based on their size compared to the membrane pore size) [30].

The results show that the membrane has a high efficiency, with a removal rate of 93% for domestic wastewater and 91% for industrial wastewater; these results are consistent with the results of [43].

In the Biological Oxygen Demand (BOD) test, the results showed a reduction of 48% for domestic wastewater and 33% for industrial wastewater, as this depends on the amount of organic matter [44], this is due to the presence of certain organic matter on the surface of the membrane, reducing the BOD percentage according to the amount of these materials present in the water and needing to be decomposed [45]. In the TPC test, the results showed a high efficiency in this regard. The number of bacterial cells decreased from 960/ml to 33/ml, and for domestic wastewater, the removal efficiency was 97%, while for industrial wastewater, the removal efficiency was 96%, as microorganisms such as bacteria attach and adhere to the membrane if the structure and the diameter of the pores are smaller than the diameter of the pores, so they adhere [46]. The results are consistent with those [47] and [48].

Table 2. The percentages of the cellulose membrane efficiency in removing pollutants from domestic wastewater.

Test	Measure unit	Before treatment	After treatment	Removal percentage
pH	-	6.7	7.5	-
Turbidity	NUT	150	20	86%
Na	mg\L	301	65	78%
K	mg\L	31	8	74%
TDS	ppm	1105	895	20%
E.C	µS/cm	2000	1016	49%
Cl-	mg\L	170	120	29%
NO_3	mg\L	2.8	2.1	25%
PO_4	mg\L	0.12	0.1	16%
TH	mg\L	375	300	20%
SO_4	mg\L	250	201	20%
TA	mg\L	166	118	30%
TSS	mg\L	442	40	90%
BOD	mg\L	18	12	33%
TPC	Cell/ml	840	27	98%

Table 3. The percentages of the cellulose membrane efficiency in removing pollutants from industrial wastewater

Test	Measure unit	Before treatment	After treatment	Removal percentage
pH	-	6.9	7.7	-
Turbidity	NUT	75	13	83%
Na	mg\L	400	75	81%
K	mg\L	33	6	82%
TDS	Ppm	1202	900	25%
E.C	µS/cm	2100	1201	43%
Cl-	mg\L	234	151	35%
NO3	mg\L	3.2	2.5	22%
PO4	mg\L	0.17	0.10	41%
TH	mg\L	1500	1001	33%
SO4	mg\L	400	300	25%
TA	mg\L	125	103	18%
TSS	mg\L	511	34	%93
BOD	mg\L	25	13	48%
TPC	Cell/ml	960	33	96%

Conclusions

The results of this study show that cellulose filters made from palm leaves can be very effective in removing various pollutants from domestic and industrial wastewater and achieve different removal rates. It is a promising and environmentally friendly method that can be widely used in wastewater treatment and purification.

Gratitude

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