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Development and comparison of membrane separation schemes for byproduct recovery from Egyptian rice straw black liquor

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Abstract

Background: Recovery of valuable ingredients from black liquor could lead to an environmentally and economically sound bioethanol production technology. In this work, two schemes comprising hybrid membrane systems incorporating ultrafiltration (UF) and nanofiltration (NF) are developed for the recovery of lignin, silica rich and cellulose/hemicellulose hydrolysates byproducts from alkaline pretreated rice straw.

Methods: The first scheme (I) comprises UF, NF and thermal vapor compression (TVC), while, the second scheme (II) includes UF, 2 stages of NF and 2 TVC units. Further treatments are suggested to produce solid byproducts with an economic value. Furthermore, material balance of the two schemes based on 1000 m³/d of black liquor and the main design features and comparative direct cost indicators of the main adopted units were deduced using WT Cost II© software.

Results: Results revealed that about (80–90%) yield of recovered byproducts from both schemes with equivalent amounts of 9.5, 5.5 and 18.5 ton/d of lignin, silica rich and cellulose/hemicellulose hydrolysates dry products, respectively. Moreover, reusable water recovery approaches 26% and 70% for schemes (I) and (II), respectively.

Conclusions: Further, the wastewater generated from scheme (II) is 2.9 times folds scheme (I) which improves the environmental impact of the former. Preliminary cost indicators revealed that both schemes have almost the same total direct capital cost.

Keywords: Black liquor, Byproducts recovery, Process design, Rice straw, Membrane

Background

Black liquor is the waste stream produced from the process of fuel production using rice straw. It contains both organic and inorganic compounds in large amounts that could be utilized in different applications. The most important applications are the pulping chemicals, adhesives, fire retardant, water purification, biodegradable plastics, and biomedical applications (Voepel et al. 2009; Li et al. 2018; Grossman and Wilfred 2019).

Traditionally, black liquor has been concentrated, then, incinerated to recover heat energy and chemicals. Although this alternative is widespread, other methods have been studied in order to remove the different types of pollutants and organic matters, these methods include: chemical and electrochemical methods, membrane filtration or adsorption (Minu et al. 2012; Hubbe et al. 2019; Lauwaert et al. 2019). Recently, different techniques were conducted for obtaining other products from black liquor with higher added value in addition to reducing operating problems, such as incrustation, corrosion and environmental problems mainly related to air pollution (Sannigrahi et al. 2010; Kevlich and Shofner 2017).

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Membrane separation technique is a promising alternative for energy generation from black liquor through the recovery of useful organic and inorganic materials (Kong et al. 2016) in addition to its impressive separation efficiency and low energy consumption over alternative strategies that rely on precipitation through acidification at higher costs (Jin et al. 2013; Jonsson et al. 2008). Owing to the high fouling potential and high viscosity of condensed black liquor, partial concentration of black liquor by membranes (up to around 30–40% of total solids) will result in a major reduction of energy consumption (Arnell et al. 2014). The various types of membranes can be functionally characterized by their molecular weight cut-off (MWCO).

Koivula et al. (2011) and Liu et al. (2014) found that the filtration capability of UF membrane was improved by conducting pre-treatment methods, such as pH adjustment, ion-exchange resin, MF or activated carbon adsorption. It was also found that micro- and ultra-filtration membranes significantly reduced the amounts of carbohydrate residuals in the separated lignin from two black liquors obtained from pulping of eucalyptus and softwood (Ziesig et al. 2014). Separating black liquor at 60 °C during the di-filtration step using a plate-and-frame polymer membrane with MWCO ranging from 6 to 50 kDa achieved an average membrane flux of 90 LMH with 25 kDa and 54% of the initial lignin concentration was recovered. It can be concluded that membranes with a higher MWCO will increase the purity of the recovered lignin, whereas membranes with a low MWCO are better if the aim is to achieve a high lignin concentration (Wallberg et al. 2003). Rejection of 94% Lignin and 97% hemicelluloses were successfully achieved from the concentration of the alkaline extraction of a sulphite pulp mill using ALFA-LAVAL—NF99HF membrane at 70 °C and a trans-membrane pressure of 13 bar with an average permeate flux of 24.2 LMH and an increase in the total solid content from 3 to 7% (*w/w*) at a volumetric concentration factor of 3:1 (Teófilo and Leitão 2016).

The extraction of lignin and hemicellulose from hardwood black liquor was performed using UF ceramic membrane (nominal cut-off 15 kDa) at 90 °C and a polymeric NF membrane (nominal cut-off 1 kDa) at 60 °C. The lignin concentration was 60 g/l in the black liquor and 165 g/l in the product stream (Jonsson et al. 2008). In another study, lignin was concentrated from 38 to 185 g/l, using UF membrane at a pressure of 207 kPa and a cross-flow velocity of 1.06 m/s. The average permeate flux was 7–21 LMH and the permeate lignin concentration was increased to 40 g/l at the end of a concentration cycle (Xiaoyuan 2016). Persson et al. (2010) concluded that UF/PES membrane recovered about 95% of the

hemicelluloses in pulping mill at flux rate ranging from 10 to 135 L MH.

In view of the limited literature on integrated membrane-based schemes for recovery of economic byproducts and water from rice straw black liquor, this work presents an endeavor toward maximizing both the byproducts and the percentage water recovery from the rice straw black liquor. Dual membrane schemes incorporating UF and NF have been investigated for the recovery of cellulose/hemicellulose hydrolysate, lignin and silica from the black liquor of alkaline pretreated rice straw which has been used for the bioethanol production.

Methods

The logical rationale for the study is based on the following:

- Development of alternative schemes components and sequences.
- Identification of each treatment scheme parameters namely % recovery and solute rejection according to specific solute molecular weight cutoff required for separation.
- Consecutive determination of each stage of treatment outputs based on the material balance considering the pre-mentioned process parameters.
- The previous steps conclude the material balance of all the scheme components.
- The data concluded is applied to the WT cost software to deduce the system technical characteristics, and direct capital cost of the scheme.

The adopted approach comprises the development of alternative schemes for the treatment of 1000 m³/d of black liquor from alkaline pretreated rice straw. These developed schemes include hybrid ultrafiltration/nanofiltration processes complemented with thermal vapor compression in addition to further treatment units to produce solid byproducts. Typical performance indicators of selected units have been developed through rationalization of the reported work. Comprehensive material balances calculations have been conducted on excel sheets for both schemes (I and II).

Comparative cost indicators related to the two schemes have been estimated on the following basis:

1. For membrane and thermal units for black liquor treatment, technical characteristics and direct capital costs were deduced using an open source software WT Cost II© developed by the Bureau of Reclamation and Moch Associates according to the design features provided. The input data was adapted to the software requirements in terms of sodium, silica and

TOC to provide simulant compositions to the actual ones taking into consideration membrane transport parameters (osmotic pressure and recovery).

2. For additional treatment units used to reach the required byproducts, the essential specifications for these treatment units and cost estimation have been developed according to the following basis:

- Purchased equipment cost were estimated from published data (Peters et al. 2004) and updated to 2021 using chemical engineering cost index
- The direct capital costs were estimated based on a multiplier to account for other items (Peters et al. 2004).

The first scheme (I) comprises UF, NF and thermal vapor compression (TVC), and additional treatment units. The second scheme (II) includes UF, 2 stage NF and 2 TVC units, and additional treatment units to process liquid streams to dry products.

Results

Developed processes description

The black liquor of alkaline pretreated rice straw based on previous lab scale experiments typically consists of: 6 g/l /cellulose hydrolysate (C/CH), 14.8 g/l hemicellulose/hemicellulose hydrolysate (HC/cellulose HCH), 10.5 g/l lignin, 8.7 g/l ashes, 6 g/l silica and 40 g/l total solids with 37.5 g/l COD and 15 g/l BOD at pH 9–10 (Sorour et al. 2021).

The developed processes units’ performance indicators were based on extensive screening and analysis of the reported experience of direct relevance to the developed schemes for recovery of organic and inorganic components from black liquor (Wallberg et al. 2003; Dafinov et al. 2005; Tolenado et al. 2010; Humpert et al. 2016; Bokhary and Mahmmoud 2017). Performance indicators for the units selected in the developed schemes are illustrated in Table 1.

The flow rates (m³/d) of permeate (Q_p) and the concentrate (Q_c) produced from each treatment step were calculated using the following equations:

$$Q_P = \Delta * Q_F \tag{1}$$

$$Q_C = (1 - \Delta) * Q_F \tag{2}$$

$$\Delta = Q_P/Q_F \tag{3}$$

where Q_F is the feed flow rate and Δ represents the membrane recovery.

The concentration of the different constituents (mg/l) in the feed (C_F), permeate (C_P) and reject (C_R) of each of the membrane units was calculated according to the following equations:

$$C_R = (C_F - \Delta * C_F * (1 - R))/(1 - \Delta) \tag{4}$$

$$C_P = C_F * (1 - R) \tag{5}$$

where R represents the membrane rejection (%).

Produced byproducts and liquid streams

Amounts of organics and inorganics in different streams of the two developed schemes were determined by overall mass balance calculations based on 1000 m³/d of black liquor from alkaline pretreated rice straw. The mass balance calculations are based on performance indicators and main technical specifications for the developed processes as depicted from previous endeavors and compiled in Table 1.

Scheme (I), presented in Fig. 1a, shows that most of the organic components (including cellulose, hemicelluloses and their hydrolysates and lignin) will be separated by UF and NF systems while, most of the silica will be separated by the thermal vapor compression unit. Scheme (II) allows further concentration of NF1 permeate stream (low organic and high silica contents) and most of the silica will be separated by the second tight NF unit which is followed by the second thermal vapor compression unit (TVC2) as shown in the process flow diagram (Fig. 1b).

Table 1 Performance indicators for the selected units in the developed schemes

	UF	NF1	NF2	TVC1	TVC2	PF1	PF2	MF
MWCO	10,000	1000	300–500					0.1–0.2 μm
Recovery (%)	80	60	60	80	80	85	85	85
Main constituents	Rejection (%)							
C/CH	50	90	90	100		5	85	5
HC/HCH	50	90	90			5	85	5
Lignin	90	90	90			90	85	5
Silica	1	5	90			10	7	95

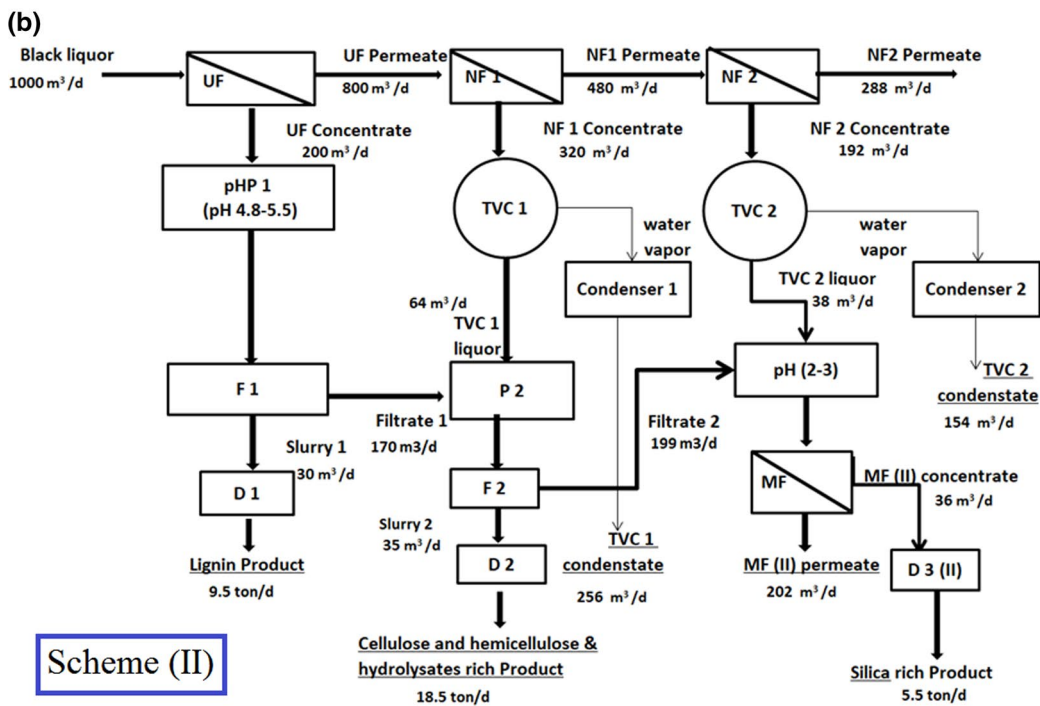
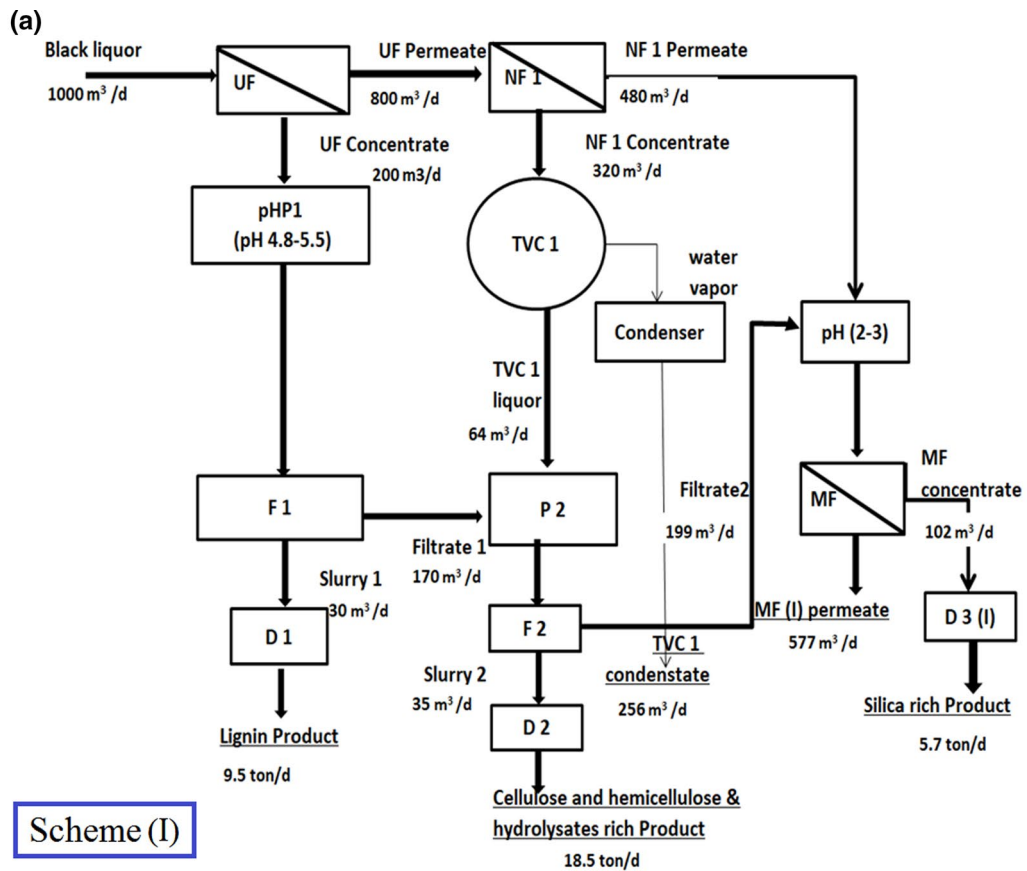


Fig. 1 Developed schemes (I) and (II) for byproducts recovery from rice straw black liquor

Scheme (II) allows minimization of the final wastewater. Further processing will achieve valuable products (lignin, cellulose/ hemicellulose hydrolysates and silica). The characteristics of initial, intermediate and final streams have been summarized in Table 2. Furthermore, the recovered byproducts from both schemes based on 1000 m³ black liquor have been illustrated in Table 3.

Design features

WT Cost II© software was adopted to identify the main design features of essential components for the developed schemes (I and II) in the main treatment scheme including UF, NF and TVC units. Moreover, the main design features of additional units for byproducts and liquids recovery were determined using chemical

engineering design methods (Peters et al. 2004) and presented in Table 4.

Comparative cost indicators

The developed schemes could provide an economic pathway for improving bioethanol production economics through the following:

1. Producing valuable materials that could participate in covering the expenses of pretreatment of lignocellulose wastes used for bioethanol production.
2. Improving water consumption in the process.

Table 2 Stream’s quantities and composition in the developed schemes

Unit	Stream	Quantity (m ³ /d)	Composition (%) (w/v)			
			C/CH	HC/HCH	Lignin	Silica
UF	Black liquor	1000	0.60	1.48	1.05	0.61
	UF permeate	800	0.30	0.74	0.11	0.60
	UF concentrate	200	1.81	4.45	4.85	0.63
NF1	NF1 permeate	480	0.03	0.07	0.01	0.57
	NF1 concentrate	320	0.71	1.74	0.25	0.65
TVC1	TVC1 condensate	256				
	TVC1 liquor	64	3.54	8.71	1.24	3.25
NF2	NF2 permeate	288	0.00	0.01	0.00	0.06
	NF2 concentrate	192	0.07	0.17	0.02	1.35
TVC2	TVC2 condensate	154				
	TVC2 liquor	38	0.35	0.87	0.12	6.74
PF1	Filtrate 1	170	2.02	4.97	0.57	0.67
	Slurry 1	30	0.60	1.48	29.09	0.42
PF2	Filtrate 2	199	0.43	1.06	0.13	1.51
	Slurry 2	35	13.81	33.97	4.27	0.64
MF (I)	MF (I) permeate	577	0.16	0.41	0.05	0.05
	MF (I) concentrate	102	0.05	0.12	0.02	5.36
MF (II)	MF (II) permeate	202	0.47	1.15	0.15	0.14
	MF (II) concentrate	36	0.14	0.34	0.04	14.90

Table 3 Recovered byproducts from both schemes based on 1000 m³ black liquor

Byproduct	ton/d*	Components (ton/d)				Yield (%)
		C/CH	HC/HCH	Lignin	Silica	
Lignin product (I) and (II)	9.5	0.18	0.44	8.73	0.13	83
Cellulose and hemicellulose and hydrolysates rich product (I) and (II)	18.5	4.85	11.92	1.50	0.23	80
Silica rich product (I)	5.7	0.05	0.12	0.02	5.46	90
Silica rich product (II)	5.5	0.05	0.12	0.02	5.30	87

*Dry weight basis

Table 4 Design features of main and additional units in the developed schemes

Item	Specifications
UF	Element flow: 38 l/s, elements/module: 90, design feed pressure: 207 kPa, membrane life: 10 years
NF softening membranes	Element flow: 30 m ³ /d, pressure drop 414 kPa, feed pressure 2760 (NF1), 2220 (NF2) kPa., No. of elements: 60 (NF1), 12 (NF2), No. of pressure vessels: 10 (NF1), 2(NF2), fouling factor: 1
TVC	Power cycle: CCGT, boiler efficiency: 95%, power consumption: 2.2 kWh/m ³ , distiller performance ratio (GOR): 8
Tanks*	Filling volume: 80%, retention time: 15 min, mixers: HDPE, volume: 3–11 m ³ , carbon steel
Filters	Centrifugal continuous bowl, diameter: 0.42 m, carbon steel
Dryers	Spray dryers, evaporation rates: 0.23–1.3 kg/s, carbon steel

*pH adjustment and precipitation tanks

3. Dramatic reduction of wastes directed for treatment and disposal and consequently reduction of pollution mitigation measures and expenses.

Comparative cost indicators related to the two schemes could be classified as follows:

1. Membrane and thermal units for black liquor treatment (main units)
2. Additional treatment units to reach the required byproducts (additional units) and wastewater treatment (WWT).

Table 5 presents Total direct costs for the main and additional units for the developed schemes based on 1000 m³/d of rice straw black liquor. The results show that the direct costs for the main processing stream are \$1,823,000 and \$2,444,000 for schemes (I) and (II), respectively. The direct capital costs for the additional unit are \$2,332,000 and \$1,665,000 for schemes (I) and (II), respectively. Finally, the total direct costs are \$4,155,000 and \$4,109,000 for schemes (I) and (II), respectively.

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Discussion

Stream feed and product quantities of the developed schemes are based on 1000 m³/d of black liquor as shown in Fig. 1. Figure 1a presents scheme I in which, the black liquor is fed to a polymeric UF membrane system (MWCO 10 kDa) for separation of lignin in UF concentrated stream while, cellulose/hemicelluloses and their

Table 5 Total direct costs for the main and additional units in the developed schemes based on 1000 m³/d of rice straw black liquor

Unit	Total direct capital cost (\$)	
	Scheme (I)	Scheme (II)
<i>Main units</i>		
UF	409,000	409,000
NF1	366,000	366,000
NF2	–	249,000
TVC1	718,000	718,000
TVC2	–	529,000
MF1	330,000	–
MF2	–	173,000
	1,823,000	2,444,000
<i>Additional units</i>		
pHP 1	40,000	40,000
P2	46,000	46,000
pH	76,000	46,000
F1	109,500	109,500
F2	109,500	109,500
D1	301,000	301,000
D2	287,000	287,000
D3	763,000	326,000
WWT	600,000	400,000
	2,332,000	1,665,000
Total scheme	4,155,000	4,109,000

Bold values indicate subtotal

hydrolysates and silica in UF permeate side were directed to NF1 membrane unit (MWCO 1 kDa) for further separation of silica in permeate stream. Cellulose, hemicellulose and their hydrolysate from NF1 concentrate were further concentrated using TVC1 unit. In scheme (II) as shown in Fig. 1b, the permeate from NF1 is directly fed to a second NF tight membrane unit (NF2) (MWCO 300–500 nm) and second TVC unit (TVC2) for further silica separation. Polymeric UF, NF1 and NF2 membranes were developed to perform at pH 9–10 and 35–40 °C. Further treatment units (pH adjustment (pH), precipitation (P)

and filtration (F), microfiltration (MF) and drying (D)) were developed to produce solid products out of the concentrated stream.

It is clear that both schemes lead to the same byproducts' quantity and quality for lignin and cellulosic and hemicellulose hydrolysate products. Silica rich by product differs slightly for scheme (II). The yield of lignin is 83% of its content in the black liquor, cellulose/hemicellulose hydrolysates yield is 80%, while silica rich product yield amounts to 90% and 87% for schemes (I) and (II), respectively.

Considering the produced liquid streams for both schemes, it is evident that condensates of scheme (I) and scheme (II) which represent reused water are 256 and 410 m³/d, respectively. Also, the hydraulic load of generated wastewater for scheme (I) is 577 m³/d representing 57.7% of initial black liquor while it is only 202 m³/d for scheme (II) although both have almost the same organic load. This represents environmental favorable conditions for scheme (II) over scheme (I). Further, analysis of the data reveals that, the second alternative demands less energy requirements due to the use of NF unit prior to TVC for concentrating silica rich stream.

The technical requirements for scheme (I) is less than that for scheme (II). However, the much lower hydraulic load directed to further processing is much lower in case of scheme (II) than scheme (I) which offers an advantage for scheme (II) over scheme (I).

The main separation processes direct capital costs for schemes (I) and (II) represent 43.9% and 59.5%, respectively. This could be attributed to higher concentrated streams with lower hydraulic load of scheme (II) than scheme (I). On the other hand, the direct capital costs for the additional units applied to treat recovered streams from scheme (I) and (II) are higher for scheme (I) than scheme (II) (2.44 and 1.665 million, respectively). This could be attributed to higher hydraulic load with lower concentration of streams for scheme (I) to be further processed to dry products. The total direct capital costs for the two schemes are very close (4.155 and 4.109 million, for scheme (I) and scheme (II), respectively).

The presented technical and cost assessment concluded the advantage of scheme (II) over scheme (I). Detailed assessment of the preferred scheme (scheme II) could be the subject of future research.

Conclusions

Black liquor produced from alkaline pretreated rice straw for bioethanol production could be a source of byproducts that would improve the bioethanol production economics. Two schemes are developed for processing of black liquor. Scheme (I) comprises UF, NF

and TVC, while, scheme (II) includes UF, 2 stage NF and 2 TVC units. The intermediate products from each stream are subjected to further processing to produce the final products. The comparison of the two schemes concluded the following:

- The two schemes provide almost the same quantities of solid byproducts, namely lignin rich, silica rich, cellulosic/hemicellulose hydrolysate products.
- Scheme (II) demands more technical requirements than scheme (I).
- Recycled water recovery in scheme (II) is much higher than that of scheme (I) (70% and 26%, respectively).
- The quantity of wastewater from scheme (I), is about 2.9 higher than that of scheme (II), although both are almost of the same solids load.
- Preliminary cost indicators revealed that both schemes have almost the same total direct capital costs. However, technical and cost assessment concluded that scheme (II) is the preferred alternative.
- Further work is recommended for detailed assessment of preferred scheme (scheme II) to be applied for different plant capacities and specific sites.

It is concluded that the use of membranes and recovery of byproducts represent an efficient approach for the treatment of black liquor. However, the economic and environmental factors should be thoroughly further analyzed to come up with the most feasible alternative.

Abbreviations

BOD: Biological oxygen demand; COD: Chemical oxygen demand; C_f: Concentration in feed; C_p: Concentration in permeate; C_r: Concentration in reject; kDa: Kilodaltons; LMH: L/hr/m²; Q_p: Flow rates permeate; Q_c: Flow rate of the concentrate; Q_f: Flow rate of feed; MF: Microfiltration; NF: Nanofiltration; TVC: Thermal vapor compression; MWCO: Molecular weight cut-off; TOC: Total organic carbon; UF: Ultrafiltration; WWT: Wastewater treatment.

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Authors' contributions

MM and AM: calculation's part and writing the research paper. SR and MH: preparing alternative schemes. HS and HA: reviewing and contributing in writing the research paper. All authors read and approved the final manuscript.

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Availability of data and materials

Data sharing is not applicable to this article as no datasets were generated or analyzed during the current study.

Declarations

Ethics approval and consent to participate

No formal ethics approval was required in this particular case.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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