ELABORATION AND CHARACTERIZATION OF CELLULOSIC MEMBRANES FOR EFFICIENT MOLYBDENUM REMOVAL

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In this work, a novel class of polymeric inclusion membranes (PIMs) for ion separation was developed. All synthesized membranes were composed of cellulose triacetate (CTA) modified by trioctyle phosphine oxide (TOPO), tributyle phosphate (TBP) or tricapryle ammonium (Aliquat-336) incorporated into the polymer as carrier and tris-(2-ethylhexyl) (TEHP) or 2-nitrophenyle octyle ether (NPOE) as plasticizer. The synthesized PIMs were characterized using various techniques, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The influence of the membrane nature was studied using supports with different physical characteristics (porosity, thickness, hydrophobicity). All synthesized membranes were applied to molybdenum recovery using this innovative process, supporting the determination of transfer fluxes and permeability factors. The transport flux and its efficiency depend on the chemical nature of the plasticizer. It was established that TEHP (viscosity η = 10.2 mPa.s, permittivity εr = 4.8) and NPOE (viscosity η = 16.9 mPa.s, permittivity εr = 24.2) produced the highest PIM transport of ions.

Keywords: molybdenum, cellulose triacetate, carrier, plasticizer, water purification

INTRODUCTION

Water pollution, which affects rivers, seas, groundwater and lakes, is the result of the discharge of wastewater, without treatment or inadequately treated, into natural water bodies, causing the degradation of ecosystems.[1] The pervasiveness of heavy metal contaminants in industrial effluents poses serious threats to both human well-being and environmental stability.[2] Therefore, there have been conspicuous efforts towards developing effective and environmentally friendly remediation techniques.[3-7]

In the field of extractive chemistry, one of the fundamental steps in process operations is the concentration and purification stage. This step increasingly relies on advanced techniques that best meet purity requirements. Among these techniques, those using selective membranes have seen significant development over the past decades, both in terms of the physicochemical processes involved and the industrial installations implemented.[8-10] Currently, four categories of membranes can be recognized: thick liquid membranes,[11] emulsion liquid membranes,[12] supported liquid membranes[13] and polymer inclusion membranes (PIMs).[14-25] Our research focuses on the development and characterization of new polymer inclusion membranes and the study of its molybdenum transport properties. These membranes are based on a cellulose triacetate (CTA) polymer backbone, but differ as a function of the choice of plasticizer, the nature of the carrier, and the transport mechanism. Physicochemical characterization of the membranes was performed using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA).

To test the applicability of the developed membranes, the dialysis process was carried out

in order to eliminate molybdenum species from synthetic aqueous solutions. Conventional parameters characterizing the performance of the membranes, such as transfer flux and permeability, were determined. The introduction of TOPO, TBP or Aliquate-336 carrier within the CTA polymeric matrix was noted to enhance considerably the capacity of the membranes to form complexes with molybdenum ions, while the incorporation of tris-(2-ethylhexyl) (TEHP) or 2-nitrophenyle octyle ether (NPOE) as plasticizer improved the physico-chemical and mechanical properties of the synthesized membranes.

EXPERIMENTAL

Chemicals

Cellulose triacetate (CTA, pure product), trioctyle phosphine oxide (TOPO), tricapryle ammonium chloride (Aliquat-336), tris-(2-ethylhexyl) phosphate (TEHP), tributyle phosphate (TBP) and 2-nitrophenyloctylether (2-NPOE) were purchased from Fluka Co. All reagents were used as received, without any further purification. The aqueous solutions were obtained by dissolving the different reagents in deionized water.

Membrane preparation method

The CTA membranes were prepared via phase inversion, according to the procedure reported by Sugiura *et al*.[14] In this method, 0.4 g of cellulose triacetate (CTA) was dissolved (during 4 hours) in chloroform ($CHCl_3$). Then, 0.1 mL of the plasticizer (TEHP or 2-NPOE) was added to the polymeric solution under vigorous stirring for 1 hour. Finally, 0.1 g of carrier (TOPO, TBP or Aliquat-336) was added to the homogeneous solution under moderate stirring for 1 hour. The obtained solution was transferred into a glass plate container (21 cm × 16 cm), and degassed in an ultrasonic cleaner for 10 minutes to remove air bubbles to form a homogeneous and stable solution. After degassing, the solution was cast and allowed to slowly evaporate for 24 hours. The resulting membrane was extracted by the addition of distilled water.

Membrane characterization

FTIR spectroscopy was used to observe the structural changes of the membranes by examining their characteristic bands. This technique was used to detect the presence of the bands corresponding to different functional groups in the neat CTA membrane and then in the complex polymeric membranes. The FTIR spectra were recorded with a Perkin-Elmer spectrometer (Spectrum One, Perkin, U.S.A). The instrument was calibrated before analysis using 32 scans at a resolution of 2 cm^{-1} in the wavenumber range of 4000–400 cm^{-1}.

The contact angle measurements were determined as the tangent angle of the drop with the membrane surface. Water contact angles were recorded with an OCA20 Data-Physics Instruments, using a syringe to control the droplet size. The average of five arbitrarily selected locations for each sample represents the reported contact angle measurements.

Scanning electron microscope (SEM) images of the membranes were obtained using a JOEL JSM 6360-LV Microscope, operating at 10 kV, after gold coating the samples. X-ray diffraction patterns were recorded on a Siemens diffractometer using monochromatized Cu K_α radiation.

Water treatment experiments

The cell used for water treatment experiments consisted of two compartments, made of Teflon, with a maximum filling volume of 100 mL, separated by the polymeric membrane. The feed solution was prepared from ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$), where molybdenum was transported in an anionic form as $(MoO_4)^{2-}$ and the strip compartment contained distilled water. Both the feed and strip aqueous phases were stirred at 800 rpm using a magnetic stirrer. The metal concentration was determined by samplings, at different time intervals, of aliquots (0.5 mL) from both the feed and strip solutions. They were analyzed with a JASCO-V-530 UV-Visible spectrophometer, for molybdenum quantification by Arsenazo III method. The additional reagents included H_2SO_4, $CuSO_4$, ascorbic acid, and KSCN, and the measurements were performed at a wavelength of 459 nm.[26]

Mass flux, J ($mol.cm^2.s^{-1}$), of the metal ions through the membranes, as transferred from the feed side of the membrane to the strip side, was determined as follows: $J = \Delta n/S\Delta t$, where Δn represents the variation in mole number of metal ions in the receiving solution during the reference time Δt; and S is the active surface of the membrane (9.61 cm^2). Three independent experiments were realized to determine the mean molybdenum concentration.

RESULTS AND DISCUSSION

Physical and chemical properties of prepared membranes

In Table 1, some of the characteristics of all synthesized membranes using each of the three carriers (TOPO, TBP, Aliquat-336) and plasticized by TEHP or NPOE have been listed, in comparison with those of the reference CTA and CTA + plasticizer membranes.

The results show that membrane thickness increases with the carrier content, and it depends on the nature of the carrier. As the carrier molecules are hydrophobic, the location of carrier molecules on the surface of the CTA modified

membranes should modify the contact angle, which is a parameter indicative of the wetting character of a material. Overall, the tabulated contact angle values indicate that the membranes made with TOPO, TBP, and Aliquat-336 are highly hydrophobic, in comparison with CTA.

Table 1
Physical and chemical properties of synthesized membranes

Membrane	Composition (weight (g))	Weight/area (mg/cm²)	Thickness (μm)	Water content (%)	Contact angle (º)
CTA	0.4	2.88	10	36	46.40
CTA+TEHP	0.4 / 0.4	4.536	12	12	75.80
CTA+NPOE	0.4 / 0.4	5.095	15	11	80.50
CTA+TEHP+TOPO	0.4 / 0.4 /0.2	5.652	18	6.66	85.59
CTA+NPOE+TOPO	0.4 / 0.4 /0.2	7.324	20	4.76	91.06
CTA+TEHP+TBP	0.4 / 0.4 /0.2	8.121	20	5.26	86.39
CTA+NPOE+TBP	0.4 / 0.4 /0.2	6.369	23	9.67	87.54
CTA+TEHP+Aliquat	0.4 / 0.4 /0.2	5.732	14	9.09	89.62
CTA+NPOE+Aliquat	0.4 / 0.4 /0.2	5.114	30	14.28	84.22

FTIR spectroscopy of cellulosic membranes

Figures 1 and 2 present the FTIR spectra of all synthesized membranes, using TEHP and NPOE as plasticizers and various carriers, compared to that of the neat CTA membrane. Tables 2 and 3 list the peak values recorded in the FTIR spectra of the reference CTA and CTA-Plasticizer-Carrier membranes and their corresponding functional groups.

The main feature of these spectra is an absorption band located around 1735 cm^{-1}, which is attributed to the stretching vibrations of the carbonyl group. The bands around 1216 and 1029 cm^{-1} correspond to the stretching modes of C–O single bonds. Less intense bands at 2940 and 2880 cm^{-1} are attributed to C–H bonds and the wide band detected in the 3500–3100 cm^{-1} region is attributed to the O–H bonds stretching modes.

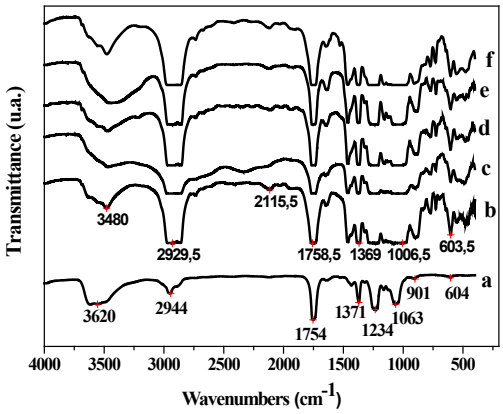


Figure 1: FTIR spectra of membranes: (a) CTA; (b) CTA-TEHP; (c) CTA-TEHP-TOPO; (d) CTA-TEHP-Aliquat-336; (e) CTA-TEHP-TBP

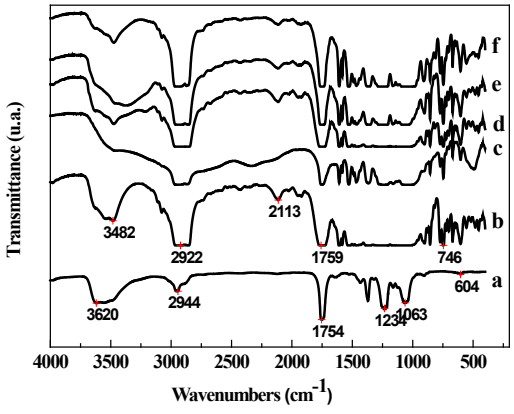


Figure 2: FTIR spectra of membranes: (a) CTA; (b) CTA-NPOE; (c) CTA-NPOE-TOPO; (d) CTA-NPOE-Aliquat-336; (e) CTA-NPOE-TBP

Table 2
Peak values and the corresponding functional groups in all membranes based on TEHP

Membranes	FTIR peaks (cm^{-1})	Assignment
CTA-TEHP	3480, 2929, 1758, 1463, 1369, 1230–1006	O–H (CTA), C–H, C=O (CTA), P–O (TEHP), -CH_3 (CTA), C–O–C (sym-asym)
CTA-TEHP-TOPO	Above mentioned + 1237	P=O (TOPO)
CTA-TEHP-TBP	Above mentioned + 1470, 1202	P–O (TBP), P=O (TBP)
CTA-TEHP-Aliquat-336	Above mentioned + 1378	N–CH_3 (Aliquat-336)

Table 3
Peak values and the corresponding functional groups in all membranes based on NPOE

Membranes	FTIR peaks (cm^{-1})	Assignment
CTA-NPOE	3482, 1759, 1525, 856	O–H (CTA), C=O (CTA), NO_2 (NPOE), C–N (NPOE)
CTA-NPOE-TOPO	Above mentioned + 1160	P=O (TOPO)
CTA-NPOE-TBP	Above mentioned + 1460, 1172	P–O (TBP), P=O (TBP)
CTA-NPOE-Aliquat-336	Above mentioned + 1367	N–CH_3 (Aliquat-336)

Two other bands characteristic of the elongation vibration bonds of P–O and P=O detected at 1470 cm^{-1} and 1202 cm^{-1}, respectively, were also observed. However, the wide band at 1367–1378 cm^{-1} confirmed the presence of the amine groups (N–C) of Aliquat-336. Overall, the obtained results showed that all the maximum values extracted from the spectrum of the CTA reference membrane, *i.e.* without plasticizer and without carrier, are present in the modified membranes' spectra as well, in addition to those characteristic of the carrier molecules, TOPO and TBP, that involve the same radicals.

Scanning electron microscopy (SEM) of cellulosic membranes

A significant aspect of membrane materials is their microstructure, which governs the dispersal of the carrier in the polymer matrix and eventually affects the membrane transport efficiency. Subsequently, substantial research effort has been dedicated to clarifying this issue. SEM provides excellent qualitative information (dense or porous membranes) and quantitative capability in measuring important subsurface features, such as porosity and layer thickness.

The morphology of the neat CTA membrane (surface view) shows that this membrane, in the absence of plasticizer and carrier, presents a porous and homogeneous structure (Fig. 3), the distribution of the pores is nearly uniform (porosity = 50%).[27] Meanwhile, all the synthesized CTA+Plasticizer+Carrier membranes present a dense structure, where the pores of the membranes have been filled by the plasticizer (NPOE or TEHP) and the carrier molecules (TOPO, TBP or Aliquat-336). Thus, these membranes are thicker and less porous.

X-ray diffraction (XRD) of cellulosic membranes

X-ray diffraction was used to characterize the membranes in order to determine the mechanism of metal ion transport. Figures 4 and 5 clearly illustrate that the developed membranes are amorphous. All synthesized membranes present a single maximum located at approximately 20° found in amorphous polymers, and may correspond to the van der Waals[28,29] "halo". Thus, the elaborated materials present basically amorphous characteristics.

The systems constituted by the mixture of CTA+Plasticizer+Carrier do not give significant diffraction peaks. It can be explained by the absence of crystallization within the elaborated membranes. In the literature, two possible mechanisms are described for explaining the diffusion of ions from the feed to the strip compartment, as a function of the crystallinity of the membrane: i) for membranes with amorphous structure: the mechanism is based on the simple diffusion through the membrane; ii) for membranes with crystalline structure: the mechanism is based on the successive complexation–decomplexation from the feed to the strip compartment by the transfer of electrons between the donor oxygen, ammonium and phosphorus of the carriers and the metal as acceptor. With the latter mechanism, the transporter molecules act as "stepping stones", and the solute moves through the membrane by "jumping" from one fixed site to another. The theory of "fixed-site jumping" was described by Cussler *et al*.[30] and Noble.[31]

Accumulation of molybdate ions at the interfaces

Figure 6 illustrates the evolution of the molar concentration of molybdenum ions on the feed-membrane and membrane-strip interfaces. A significantly different behavior of the two flow rates at the beginning of the transport can be observed. This can be attributed to the low diffusivity of the complexes inside the membrane or to a decomplexation rate on the strip side lower than the complexation rate on the feed side. It was observed clearly that the metal quantity increased in the strip phase and decreased in the feed

compartment over time. Additionally, it was also noted that a transport time of 54 hours is not sufficient to obtain equilibrium.

O. Arous *et al*.[27] and N. Abdellaoui *et al*.[32] studied the fundamental parameters governing transport through polymer inclusion membranes, including the metal concentration in the feed and strip compartments, the nature and concentration of the carrier.

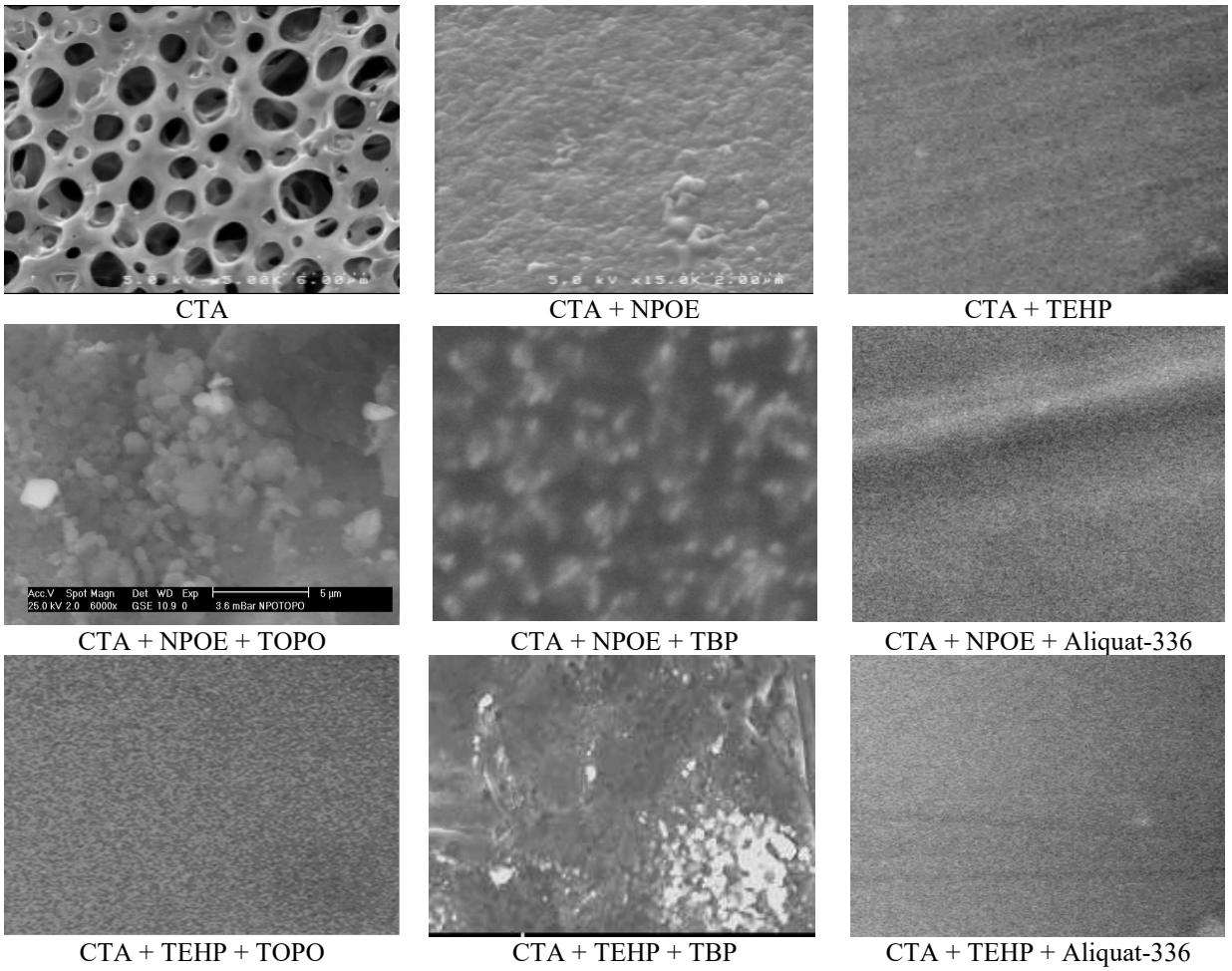


Figure 3: SEM images of all synthesized membranes

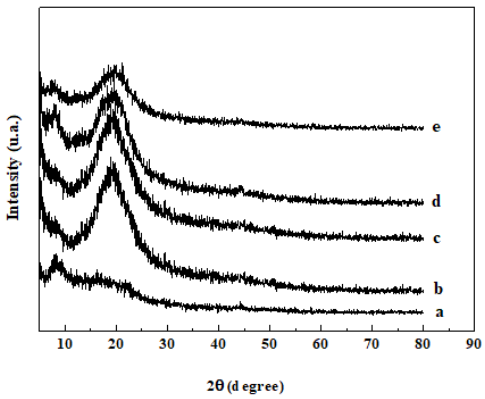


Figure 4: X-ray diffractograms of (a) CTA, (b) CTA+TEHP; (c) CTA+TEHP+TOPO, (d) CTA+TEHP+Aliquat-336, (e) CTA+TEHP+TBP membranes

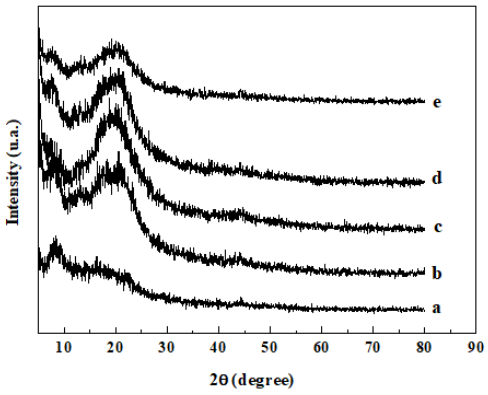


Figure 5: X-ray diffractograms of (a) CTA, (b) CTA+NPOE, (c) CTA+NPOE+TOPO, (d) CTA+NPOE+Aliquat-336, (e) CTA+NPOE+TBP membranes

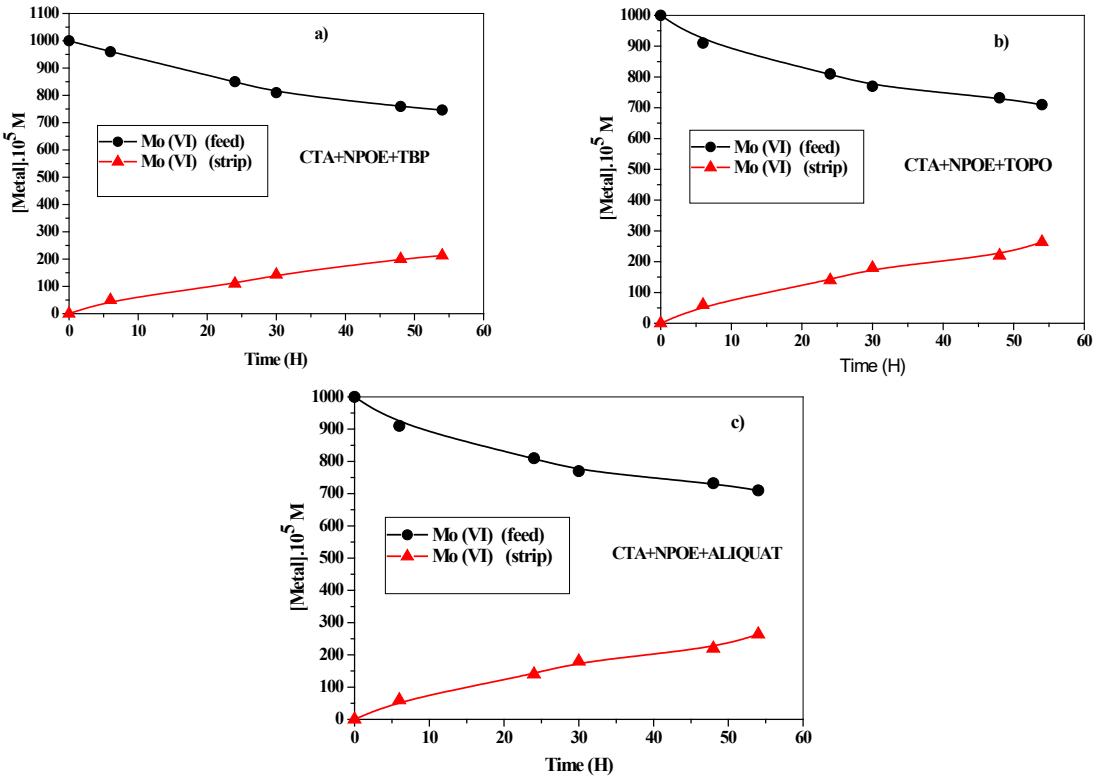
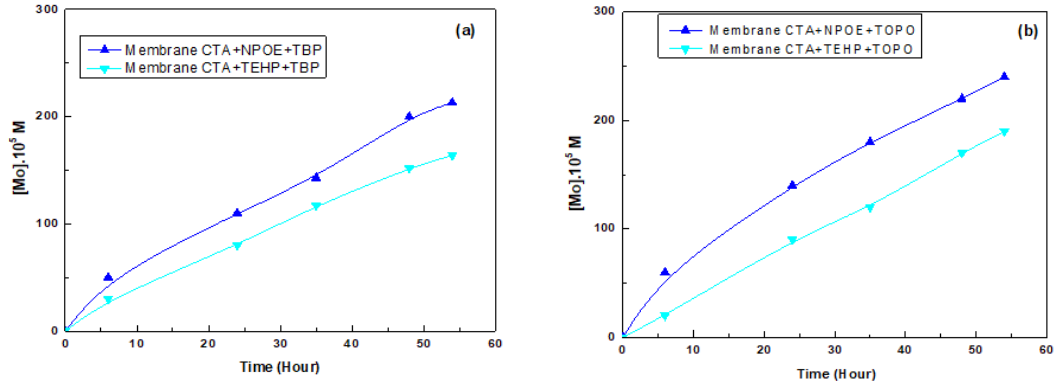


Figure 6: Evolution of molybdenum concentration in the feed and strip compartments as a function of time using the synthesized membranes: a) CTA+NPOE+TBP, b) CTA+NPOE+TOPO and c) CTA+NPOE+Aliquat-336

Influence of plasticizer on molybdenum transport

Plasticizers are well-known in polymer processing as compounds used to ensure flexibility and to avoid brittleness and cracking. In this study, two plasticizers (NPOE and TEHP) were tested in the order to verify their effect on the molybdenum transport through the PIMs. Figure 7 shows the evolution of molybdenum concentration in the strip compartment for membranes made with the two different plasticizers.

It may be remarked that all the membranes plasticized with NPOE are more efficient than those plasticized with TEHP. This suggests that the carriers are more mobile in NPOE than in TEHP, likely due to the higher solubility of the transporter in NPOE. Indeed, the plasticizer polarity influences the chemical potential of metal ion partitioning into the membrane, whereas increasing the viscosity of the plasticizer decreases the rate of transport, most likely by inhibiting the diffusion process.



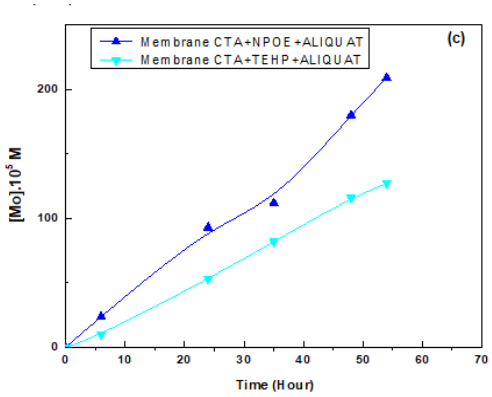


Figure 7: Evolution of molybdenum concentration in the strip compartment versus time for each of the plasticizers used (NPOE or TEHP) and carriers: a) TBP, b) TOPO, c) Aliquat-336

Table 4
Flux of molybdenum transport through polymer inclusion membranes

CTA + Plasticizer + Carrier	Flux of molybdenum (mol.cm^{-2}.s^{-1})
CTA + NPOE + TBP	12.65 10^{-10}
CTA + TEHP + TBP	09.74 10^{-10}
CTA + NPOE + TOPO	14.25 10^{-10}
CTA + TEHP + TOPO	11.28 10^{-10}
CTA + NPOE + Aliquat-336	12.41 10^{-10}
CTA + TEHP + Aliquat-336	07.54 10^{-10}

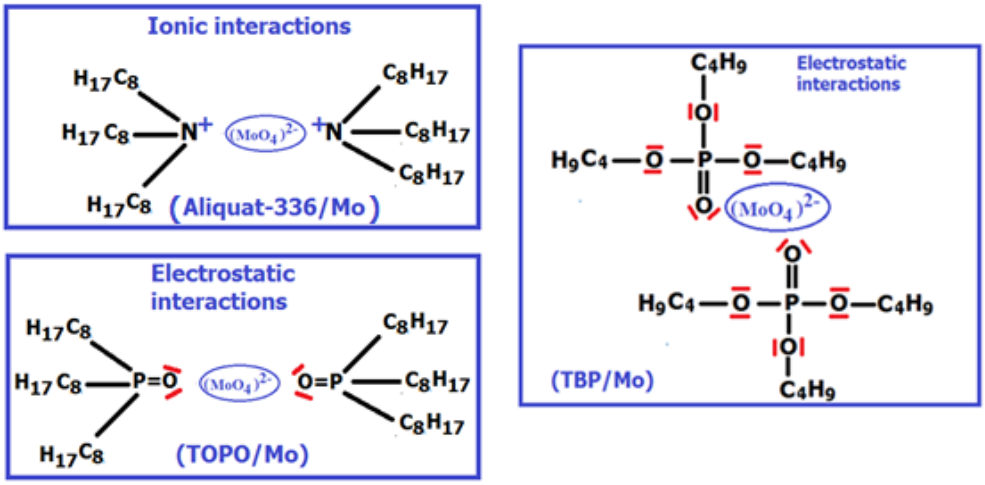


Figure 8: Ionic and electrostatic interactions developed between molybdenum ions and the three carriers: TBP, TOPO and Aliquat-336

Influence of carrier on molybdenum transport

The carriers are responsible for facilitating the transport of the target component across the membrane. The carrier can be a complexing agent, a chelating agent or an ion exchanger. The transport has been achieved using three different carriers: tributyle phosphate (TBP), trioctyle phosphine oxide (TOPO) and tricapryle chloride ammonium (Aliquat 336) respectively.

Table 4 presents the flux values for molybdenum transport achieved with the various membrane formulations prepared in this study. The results indicate that all carriers, *i.e.* TBP, TOPO and Aliquat-336, are effective for molybdenum transport. The flux values vary depending on the carrier and the plasticizer used in membrane synthesis, accentuating the importance of the transporter's nature in this facilitated process. The obtained results show that TOPO is the best carrier for molybdenum ions. The selectivity at the interface is much higher when the metallic ion is more interconnected with

the carrier by electrostatic and van der Waals interactions, in the case of TBP and TOPO, and by ionic interactions in the case of Aliquat-336 (Fig. 8).

CONCLUSION

This study developed polymer inclusion membranes based on cellulose triacetate, with various carriers and plasticizers, and examined their molybdenum transport properties. The results demonstrated that the thickness increases with the amount of carrier and depends on its nature. The contact angle values obtained with water showed that membranes based on TOPO, TBP, and Aliquat-336 are highly hydrophobic. Scanning electron microscopy revealed that the CTA + Plasticizer + Carrier membranes have a dense structure, where all the pores are filled with plasticizer and carrier. X-ray diffraction analysis indicated that the membranes have an amorphous structure. The study revealed that the membranes plasticized with NPOE are more efficient than those plasticized with TEHP. Also, the flux and permeability values indicate that the transporters TBP, TOPO, and Aliquat-336 are effective for molybdenum transport. Our results indicate that facilitated transport through plasticized membranes is an attractive and effective way to solve the enduring problem of membrane stability, whilst improving the permeability to metal ions.

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