



Article

Wet Process Phosphoric Acid Purification Using Functionalized Organic Nanofiltration Membrane

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Abstract: The utilization of phosphoric acid in various sectors, e.g., food industry, is controlled by the authorized concentration limit of impurities. However, industrial phosphoric acid is contaminated with undesirable impurities (such as F, Al, Fe, Mg, etc.). Herein, this study aimed to evaluate the efficiency of the membrane purification process of pretreated industrial phosphoric acid using a pre-modified nanofiltration membrane. We demonstrated that the prior pretreatment steps for industrial phosphoric acid allowed the elimination of sulfate, fluorine and arsenic. Further purification of the obtained pretreated phosphoric acid using membrane cells reduced the concentrations of Cd, Al, Fe and rare earth elements by 94.81, 99.30, 99.63 and 96.67%, respectively. The membrane is functionalized by a deposit of a high molecular weight polycationic polymer of polyethyleneimines in order to produce a highly charged membrane surface to enhance the separation efficiency, selectivity and stability of the membrane. We found that the purification process relies on electrostatic repulsion between the functionalized membrane and highly charged ions, and the reduction rate of metals is a cation charge-dependent parameter. The laboratory and industrial pilot scale results showed that this process allows the production of food-grade phosphoric acid.

Keywords: nanofiltration; phosphoric acid; membrane; functionalization; purification; pilot scale



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1. Introduction

Phosphoric acid (PA) is the second most produced acid worldwide after sulfuric acid [1,2]. It is an indispensable intermediate product in the synthesis of fertilizer. In addition, it is extensively used as an additive in food and detergent and by pharmaceutical industries [3]. Mainly, PA stems from the wet attack of phosphate rocks by concentrated sulfuric acid. However, industrial PA contains numerous impurities such as Fe, Al, Cu, Zn, etc. [4,5] that restrict its usage in food and by the pharmaceutical industry. Various methods have been used to eliminate or reduce these impurities. Among them, precipitation, crystallization, liquid–liquid extraction and ion exchange are the best known processes. However, the use of additives and the generation of effluents limit the application of these conventional techniques. As a result, researchers have started to direct their works toward investigations of new green and sustainable alternatives. In line with these considerations, the membrane-based process, widely used in various purification processes, seems to be very interesting as it is energy-efficient, needs no chemicals and is easy to scale up. However, one of the major limitations of this technology is the design and the development of a stable and highly selective membrane mainly under harsh conditions, i.e., high temperature and low pHs.

The precipitation method is the most economical technique, but it is less selective and generates a large amount of precipitate that requires a further separation procedure. G. Bone et al. (1986) found that PA treatment by ammonium reagents $R(NH_4)$ for 1 h at a temperature range of 50 to 70 °C favored the precipitation of $(NH_4)_x(Mg)_y(Al)_z(F,OH)_6$. X, Y and Z values depended on Mg, Al and F initial concentrations [6]. The addition of fluoride and, if necessary, aluminum ions promote the elimination of Mg and Al through $MgAl_2F_8$ and $MgAlF_5$ precipitation [7]. Fluoride and silicate ions existing as a stable complex SiF_6^{2-} in PA are precipitated in the form of Na_2SiF_6 using a sodium compound [8]. Urea also has been used to precipitate P_2O_5 in the form of urea phosphate. The precipitation process consists of attacking 30–50% P_2O_5 PA using 0.5 to 3% of urea in the presence of 2 to 7% NO_3^- . The formed precipitate contains 40% P_2O_5 , 4% NO_3^- , 1% urea, 41 ppm Fe, 1 ppm As, 2 ppm Pb, 9 ppm Mg, 10 ppm F, 0.05 ppm SO_4^{2-} and 9 ppm Ca. Then a gas mixture of $NO + NO_2$ and air is used to treat the precipitate at a volume ratio of nitrous/air = 108/300 to eliminate excess urea [9].

The purification of PA by crystallization and solvent extraction allows reduction of the amount of Mg, Fe and F [10]. The crystallization is not suitable for industrial scale applications, since it partially purifies the PA and must be accompanied by the addition of oxidants to remove impurities such as calcium phosphate, chromium, cadmium and manganese [11]. However, other crystallization processes have been developed to avoid the use of additives. The technique relies on PA (60 to 66% P_2O_5) cooling under -5 °C and then continuously feeding a crystallization container to prepare the crystals. Results demonstrated a high performance for this process since more than 90% of metallic (Fe, Al, Mg, etc.) and non-metallic (F, SO_4^{2-} , etc.) impurities were removed [12].

Currently, phosphate plant uses the solvent extraction process to purify PA. A mixture of Tri-n-butyl phosphate (TBP) and isopropyl ether (IPE) is applied in a counter-current process. The solvent mixture, containing 95% TBP and 5% IPE, is used within a volume ratio of solvents:PA = 8, at a temperature varying between 5 and 10 °C. Afterwards, the solvent is discharged and recycled with water at counter-current [13].

Ion exchange resins have also been studied to extract lanthanides. Literature results demonstrate that rare earth elements (REEs) are removed from PA and phosphogypsum by resin treatment during phosphoric acid manufacture using cationic resins with a sulfonic acid function (DOWEX C_500) [14,15].

Reverse osmosis (RO) and nanofiltration (NF) are downstream finishing processes [2]. Membrane technology has newly emerged in the phosphate sector to purify acid and reduce metallic ion impurities without reducing the PA concentration [16,17]. González et al. (2002) demonstrated that reverse osmosis is effective at concentrations lower than 2 mol/L, while nanofiltration has good performance at concentrations up to 8 mol/L. The process of purification is based on the rejection of ions by the membrane according to the ion-exclusion model, while the small neutral molecules pass through the membrane [2]. The examination of commercial nanofiltration membranes shows that Desal KH membrane can retain 92–95% of copper [18].

This work presents the purification of industrial PA obtained from Jorf Lasfar PA plant using membrane technology. Impurity removal using the membrane relies on the ionic charge-dependent process governed by the electrostatic repulsion between a functionalized membrane (positively charged) and cationic ions, rather than the membrane porous size. Unlike the discussed technologies, this process relies on the reduction of the totality of undesirable impurities.

The purification was carried out on a laboratory and industrial pilot scale in two steps:

- Purification of PA by membrane treatment.
- Concentration of treated PA up to 54% P_2O_5 .

2. Materials and Methods

The PA (25% P_2O_5) studied in this work was provided from the Moroccan phosphoric acid plant in Jorf Lasfar. The purification process consisted of two major steps:

1. PA purification by a modified nanofiltration membrane to remove impurities;
2. Concentration of purified PA by evaporation.

Copper chloride dihydrate with a purity $\geq 99.0\%$, VWR Chemicals, was used to prepare the aqueous solution of Cu^{2+} used for membrane stability monitoring.

The MPS36 membrane used in the purification process was a commercial Koch membrane system. Polyethyleneimines (PEI) Zetag IP 2M were purchased from BASF. PEI are water-soluble multifunctional cationic polyethyleneimines with a branched polymer structure [19]. The average molecular weight of polyethyleneimines (PEI) Zetag IP 2M is 2,000,000 g/mol. However, it was reported that PEI are thermally stable and start to decompose in air at 250 °C [20].

Firstly, the membrane was washed with ethanol for at least 1 h, then compressed at 40 bar prior to the modification step. The functionalization of the nanofiltration membrane MPS36 (4 inch spiral diameter with a molecular weight cut off (MWCO) of 1000 g/mol) chemically stable in concentrated PA was conducted by a deposit of a high molecular weight polycationic polymer of PEI. The adsorption step was carried out at a temperature of 60 °C under 10 bar in a solution of H_3PO_4 0.1 mol/L containing 250 ppm polyethyleneimines.

Laboratory tests were performed using a frontal filtration cell. In each test, 300 mL of PA was treated under a mechanical agitation of 200 RPM. The membrane surface area is 43 cm².

The lab-scale pilot tests were executed on a cross-flow filtration cell. The membrane chamber in the NF cell is made of spiral 316 L stainless steel. NF standard circulation speed and transmembrane pressure are up to 1700 L/h and 40 bar, respectively. The pilot alimentation capacity is 100 L and the membrane surface is 1.6 m². Table 1 summarizes the main parameters of the pilot instrument, and Figure 1 shows a schematic representation of the experimental setup.

Table 1. Characteristics of the nanofiltration pilot.

| Characteristics | Filtration Pilot |
|------------------------------------|------------------|
| Membrane surface (m ²) | 1.6 |
| Alimentation capacity (L) | 100 |
| Pressure (bar) | Up to 40 |
| Flow rate (L/h) | Up to 1700 |

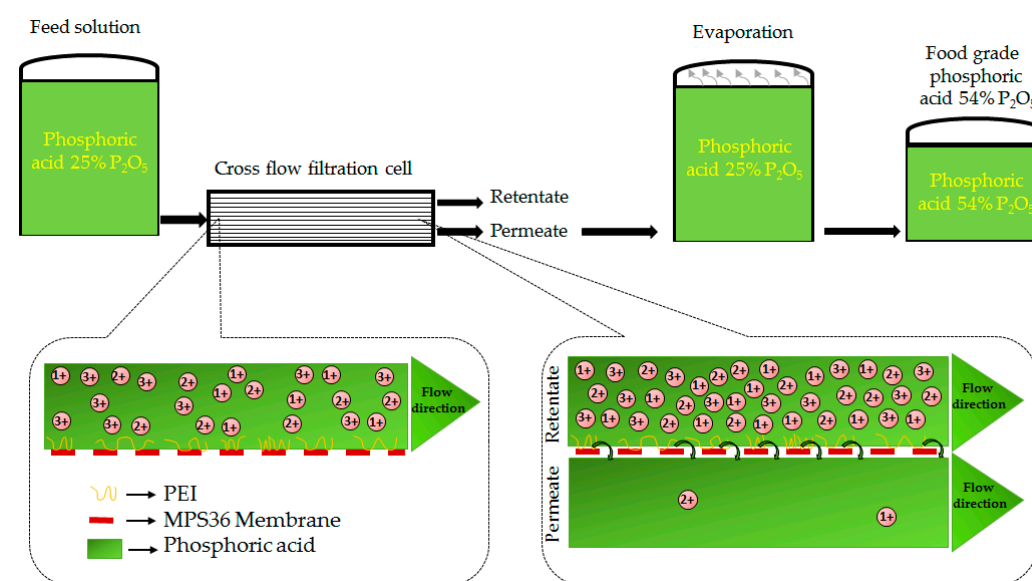


Figure 1. Schematic representation of the purification process.

The reduction rate of each element (Abt) is calculated by the follow Equation (1):

$$Abt (\%) = \left(1 - \frac{C_p}{C_i}\right) \times 100 \quad (1)$$

where, C_p and C_i are the concentrations of impurities in the permeate and raw solutions, respectively.

The selectivity (S) of the membrane is calculated by the Equation (2):

$$S = \frac{(100 - Abt_{solvent})}{(100 - Abt_{solute})} \quad (2)$$

where, $Abt_{solvent}$ and Abt_{solute} are the reduction rate of the solvent (phosphoric acid) and the reduction rate of impurities (i.e., Al, V, Cd, Cu, SO_4^{2-} and F^-), respectively.

The factor of volume reduction (FVR) is calculated using the Equation (3):

$$FVR = \frac{V_0}{(V_0 - V_{Permeate(t)})} = \frac{V_0}{V_{retentate(t)}} \quad (3)$$

where, V_0 , $V_{permeate(t)}$ and $V_{retentate(t)}$ are the initial volume of PA, the volume of permeate and the volume of retentate at a moment (t), respectively.

Inductively Coupled Plasma Mass Spectroscopy ICP-MS (PerkinElmer NexION 300X, Waltham, MA, USA) was used for REEs determination, while Inductively Coupled Plasma Atomic Emission Spectroscopy ICP-OES (Thermo Jarrell Ash IRIS/AP) was used for Al, Fe, Ca, Mg, K, Na, Cd, Co, Cr, Cu, Mn, Mo, V, Zn and As determination. The content of phosphorus present in the PA was determined by Ultraviolet UV (SHIMADZU UV-1900) using vanadomolybdate. Sulfates were determined by volumetric method using sulfonazo III and barium chloride. The fluoride concentration was measured with a selective ion electrode (METTLER TOLEDO) for fluoride analysis, and silicon analysis was accomplished by Atomic Absorption Spectroscopy AAS (PerkinElmer PinAAcle 900T). All analyses were realized at the UM6P-Jorf Lasfar chemical analysis laboratory.

3. Results and Discussion

3.1. The Effect of PEI on Membrane Permeability and Selectivity

3.1.1. Analysis of Membrane Selectivity

The development of the PA treatment process should consider the properties of PA produced from the PA plant. This facilitates the incorporation of the scale-up process in the PA industry. In line with this consideration, the filtration process was carried out at 60 °C since the PA plant generates PA with a temperature of 60 °C. Hence, the tests here were performed at 60 °C to ensure the viability of the process at this temperature.

PA treatment at 60 °C, 30 bar pressure and stirring speed of 200 RPM using functionalized and non-functionalized membranes (Figure 2) indicated that membrane modification using PEI showed a high selectivity toward aluminum, vanadium, cadmium and copper ions, which confirmed the efficiency of PEI adsorption on MPS36. The selectivity of Al, V, Cu and Cd was folded by 123.73, 131.45, 46.56 and 18.53, respectively, using a modified membrane, while the selectivity toward sulfate and fluoride ions was very low, i.e., 1.06 and 1.86, respectively. Gao et al. (2016) measured the zeta potential of hollow fiber organic membranes and showed that PEI treatment affected the surface charge of membranes. In high acidic media, it was found that the surface charge of modified membranes was highly positive [21]. Herein, the selectivity enhancement was due to the deposition of PEI on the membrane surface, which favored the increase of surface electrostatic repulsion between the cationic impurities present in PA and the cationic charge of the polymer.

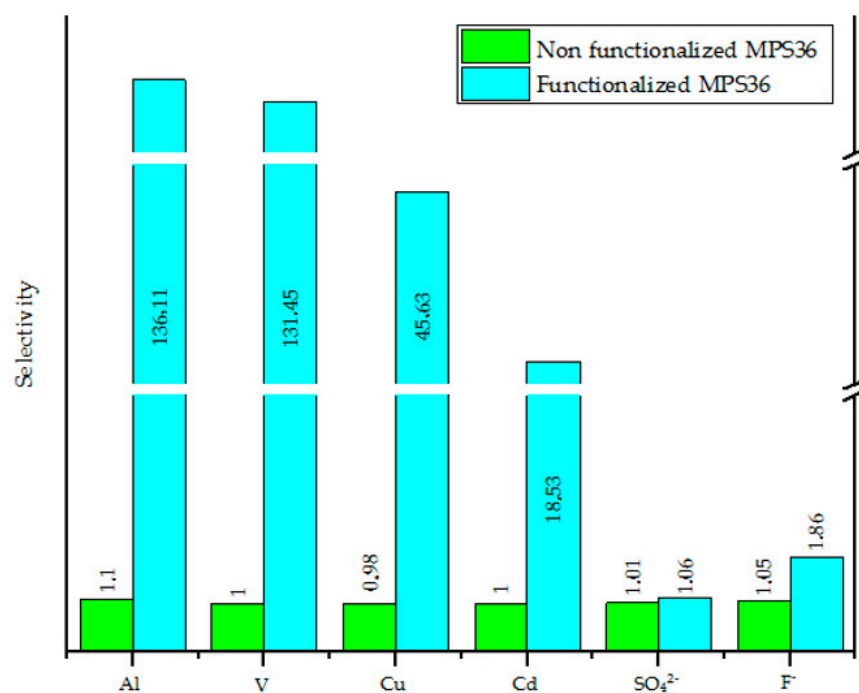


Figure 2. Selectivity of MPS36 and functionalized MPS36.

3.1.2. Analysis of Membrane Permeability

Membrane performance was analyzed in terms of PA permeability under a pressure of 30 bar and stirring speed of 200 RPM at 60 °C (Figure 3). The permeability of the functionalized membrane has decreased compared to the non-functionalized membrane. The decrease in permeability confirmed the presence of PEI on the membrane. However, the permeability of the non-functionalized membrane changed drastically by the FVR changes. The permeability of the functionalized membrane changed slightly by the FVR changes, which implied the stability of the functionalized membrane during PA treatment.

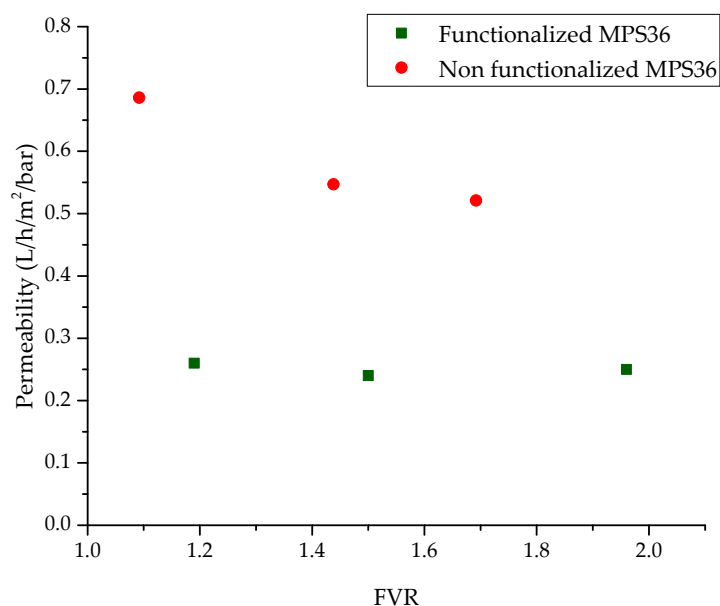


Figure 3. The permeability variation of MPS36 and functionalized MPS36 versus the factor of volume reduction (FVR).

3.2. Stability of Functionalized Membrane

The functionalized membrane was conserved in PA 25% to evaluate its stability in the high acidic media of PA. Afterwards, the functionalized membrane was used to purify an aqueous solution, pH = 5.5, of copper chloride dihydrate (0.05 g/L). Figure 4 shows that copper reduction remained stable at $96 \pm 1\%$ up to 350 days. This implied the high resistivity of the functionalized membrane to the high PA acidity and the irreversible adsorption of PEI on the membrane.

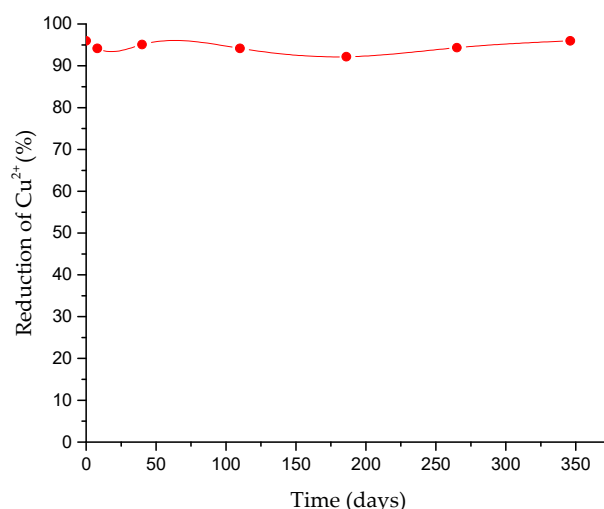


Figure 4. The variation in copper reduction by the functionalized MPS36 over time at 10 bar pressure, 200 RPM and ambient temperature.

In addition, regarding the water permeability variation over time (Figure 5), it was found that the permeability of the functionalized-conserved membrane remained stable for the entire time range of 270 days. The functionalized membrane had a steady flow of 3.5 ± 0.2 L/h/m²/bar, which confirmed the non-degradability of the membrane and PEI in PA. This indicated that the porous of membrane, rather than PEI selectivity toward cationic ions, was not affected by PA acidity.

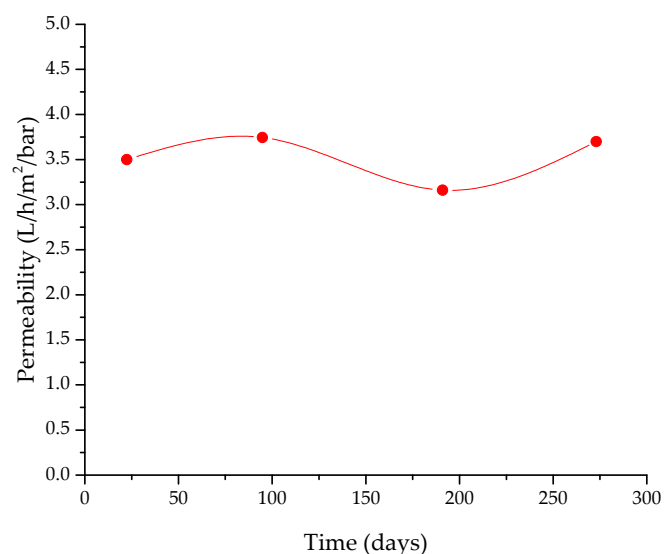


Figure 5. Water permeability of functionalized MPS36 over time.

3.3. Purification of Phosphoric Acid

In order to maintain the high efficiency and to extend the lifetime of the modified membrane it is necessary to pretreat the industrial PA to avoid the clogging of the porous membrane. Numerous methods have been used to desulfate industrial PA, e.g., the utilization of BaCO_3 [22], $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ [4] or natural material consisting mainly of calcite ($\text{Ca}_{0.97}\text{Mg}_{0.03}\text{CO}_3$), quartz (SiO_2) and a low clay fraction (illite) [23]. The dearsenification is carried out through arsenic precipitation as arsenic sulfide, arsenic removal by electrodeposition [24] or by a vertical zone melting technique [25]. In addition, it was reported that the minimization of the PA content of fluoride can be achieved by PA treatment using silicon dioxide followed by the addition of sodium carbonate [26], SiF_4 formation by adding silicon dioxide at a high temperature [4] or amine-based extractants [27]. El-Asmy et al. (2008) found that PA clarification by removing humic acid and suspended materials is attained by bentonite clay usage [26]. However, Moroccan PA undergoes a desulfation, dearsenification, defluorination and clarification at the PA production plant. The desulfation, dearsenification, defluorination and clarification are carried out by adding natural Moroccan phosphate rock, sodium hydrosulfide, sodium carbonate and bentonite clays, respectively.

The quality of PA depends on impurities in the phosphate ore [28,29]. The mineral composition present in the PA was determined (Table 2). The major element contents in PA are phosphorus, sulfate, aluminum, iron, calcium, magnesium, potassium, sodium, silica, and fluoride. In addition, other trace elements such as cadmium, cobalt, chrome, copper, manganese, molybdenum, vanadium, zinc, arsenic and rare earth elements are present at low concentrations, i.e., between 0.79 and 285 ppm.

Table 2. Elemental content of raw PA, pretreated PA and nanofiltrated PA at 60 °C and 600 L/h flow rate.

| Elements | Raw PA | Pretreated PA | Final Product | Reduction (%) |
|----------------------------|--------|---------------|---------------|---------------|
| P_2O_5 (%) | 25.90 | 25.83 | 25.00 | 3.21 |
| SO_4^{2-} (%) | 2.05 | 0.01 | 0.009 | 10.00 |
| Al (%) | 0.50 | 0.43 | 0.003 | 99.30 |
| Fe (%) | 0.28 | 0.27 | 0.001 | 99.63 |
| Ca (%) | 0.23 | 0.25 | 0.020 | 92.00 |
| Mg (%) | 0.39 | 0.41 | 0.004 | 99.02 |
| K (%) | 0.04 | - | 0.007 | 82.50 |
| Na (%) | 0.10 | - | 0.030 | 70.00 |
| Si (%) | 1.02 | - | 0.090 | 91.18 |
| F (%) | 1.57 | 0.10 | 0.050 | 50.00 |
| Cd (ppm) | 15.00 | 16.00 | 0.830 | 94.81 |
| Co (ppm) | 0.79 | 0.79 | 0.002 | 99.75 |
| Cr (ppm) | 240.00 | 215.00 | 0.780 | 99.64 |
| Cu (ppm) | 30.00 | 30.00 | 0.640 | 97.87 |
| Mn (ppm) | 14.00 | 14.00 | 0.150 | 98.93 |
| Mo (ppm) | 4.00 | 4.00 | 0.170 | 95.75 |
| V (ppm) | 208.00 | 187.00 | 1.350 | 99.28 |
| Zn (ppm) | 285.00 | 285.00 | 3.490 | 98.78 |
| As (ppm) | 17.00 | 3.00 | 3.00 | 0.00 |
| REEs (ppm) | 60.00 | 60.00 | 2.00 | 96.67 |

The relative error of elemental concentration was less than 5%.

PA pretreatment prior to membrane purification allowed having a clarified solution in which the concentration of sulfate, fluoride and arsenic decreased by 205, 15.7 and 5.6 fold, respectively, compared to raw PA. The measured absorbance of the clarified solution was 0.143 at $\lambda = 408$ nm, which explained the strong elimination of suspended matter that increased the clogging of the membrane and decreased its performance.

Table 2 clearly shows that the cross-flow purification of PA at the lab-scale pilot with the functionalized membrane allowed reaching a reduction rate above 99% for trivalent metallic ions of aluminum and iron, more than 96% for REEs, and more than 92% for divalent metallic ions such as calcium, cadmium, copper and magnesium. However, monovalent ions showed lesser reduction rates of 82.5 and 70% for potassium and sodium, respectively.

In general, the transfer of ions through the membrane is controlled by the membrane charge rather than pore size. Sun et al. (2011) demonstrated that PEI modification of organic membranes reduces the membrane pore size and changes the surface charge of membranes to positive values in high acidic media [30]. However, the low reduction rates of negatively charged ions of SO_4^{2-} and F^- , i.e., 10 and 50%, respectively, confirmed the dominance of electrostatic repulsion phenomena over steric effect. Herein, since the surface of the membrane is positively charged, it induced rejection of highly charged cations by electrostatic repulsion, which may explain the higher reduction rate of trivalent rather than divalent and monovalent cations, respectively. It is well-known that iron exists in PA as $\text{Fe}(\text{HPO}_4)^+$, $\text{Fe}(\text{H}_2\text{PO}_4)^{2+}$ and $\text{Fe}(\text{H}_2\text{PO}_4)_4^-$ species [31]. According to the results, the overall reduction efficiency of iron was 99.63%, indicating that iron phosphate complexes of $\text{Fe}(\text{HPO}_4)^+$ and $\text{Fe}(\text{H}_2\text{PO}_4)^{2+}$ are more predominant than $\text{Fe}(\text{H}_2\text{PO}_4)_4^-$. Moreover, based on the obtained results, we also expected that cadmium may exist in its free aquo ionic form within $\text{Cd}(\text{H}_2\text{PO}_4)^+$ [32]. In addition, G. Senanayake et al. (2016) reported that neodymium (Nd) forms complexes of $\text{Nd}(\text{HPO}_4)_2^-$, $\text{Nd}(\text{H}_2\text{PO}_4)^{2+}$ and $\text{Nd}(\text{HPO}_4)^+$ in phosphoric acid [33–35]. However, neodymium was used as a representing of REEs, as REEs exhibit similar chemical and physical properties [36]. In line with obtained results, the predominant REEs forms in PA are more likely $\text{RE}(\text{H}_2\text{PO}_4)^{2+}$ and $\text{RE}(\text{HPO}_4)^+$.

3.4. Concentration Polarization

Concentration polarization restrains remarkably membrane performance, as it affects the permselectivity [37] and favors the decline of flux [38] during the purification process. Herein, to assess the effect of the concentration polarization we monitored the concentration of iron (IC) as a function of time. Results in Figure 6 demonstrated that IC increased significantly in the retentate after 13 h. The instantaneous monitoring of the permeate showed that IC increased drastically after 13 h, indicating the start of concentration polarization. Thus hinder the electrostatic repulsion of iron. However, the authorized concentration limit accepted for food-grade PA is 20 ppm [39]. The screening of IC in the cumulative permeate indicated that IC remained below the authorized concentration limit and reached a critical value of 20.1 ppm after 22 h. Hence, the optimal operating time adopted for the purification process was 22 h.

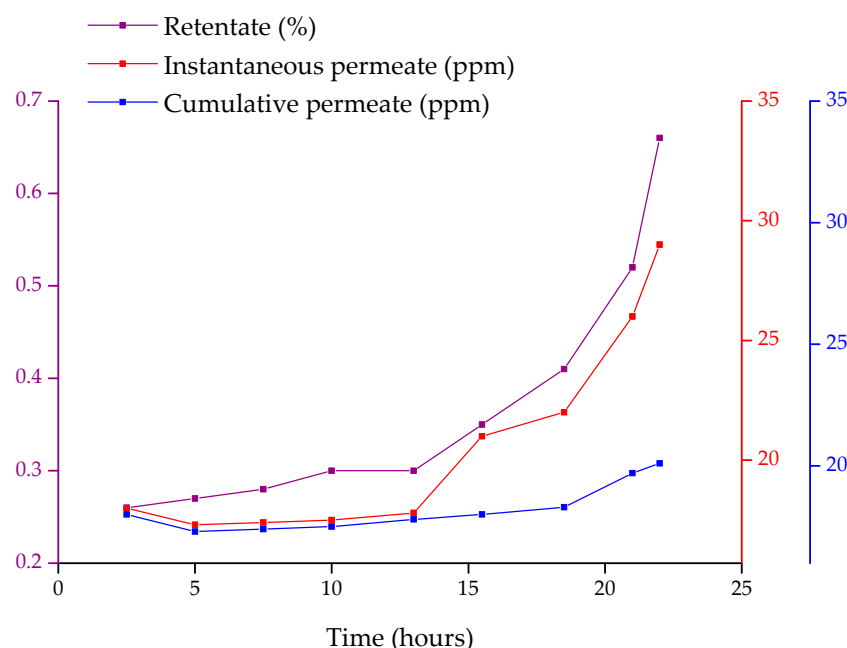


Figure 6. The variation in iron concentration in retentate, instantaneous permeate and cumulative permeate as a function of time.

3.5. Evaporation of Purified Phosphoric Acid

The evaporation of purified PA (25% P₂O₅) allowed the concentration and the preparation of high quality PA (54% P₂O₅). The concentration was achieved without changing the acid color or distressing its quality. Elemental analysis (Table 3) showed that the metallic and nonmetallic elements in purified concentrated PA were low. Al, Fe, Mg, F and As were not detected in purified concentrated PA, while Na, Cd and Cr concentrations were 0.06%, 2 ppm and 1.5 ppm, respectively. The upgrade of phosphoric acid to specified levels of As, Fe, F, Mg and Cr content lower than 3, 20, 10, 20 and 2 ppm, respectively, for food-grade quality was achieved [24,39,40].

Table 3. Purified concentrated phosphoric acid composition.

| Elements | Purified Concentrated PA 54% P ₂ O ₅ |
|-----------------------------------|--|
| P ₂ O ₅ (%) | 54.55 |
| Fe (%) | ND |
| Al (%) | ND |
| Mg (%) | ND |
| Na (%) | 0.06 |
| F (%) | ND |
| Cr (ppm) | 1.50 |
| Cd (ppm) | 2.00 |
| As (ppm) | ND |

ND: not detected.

3.6. Industrial Pilot Scale

To evaluate the quality of PA produced by the NF membrane process on a high scale level, an industrial pilot scale unit with a production capacity of 160 L/h was built. The raw PA 25% P₂O₅, industrial grade, was subjected to the same steps as in the laboratory-scale procedure, leading to the food-grade PA 54% P₂O₅. The nanofiltration pilot operated for 24 h per day. After 22 h of PA purification, a cycle of 2 h membrane cleaning was required to maintain the functionality of the membrane. The membrane cleaning was executed using a solution of 1 g/L EDTA at ambient temperature under a pressure of 10 bar.

Results in Table 4 demonstrated that metals reduction at the industrial pilot unit were slightly higher than laboratory pilot scale. However, we observed that the reduction of metals depended on the cationic charge of metals within the same pattern noticed at laboratory scale.

Table 4. Elemental content of nanofiltrated PA at the industrial pilot scale unit.

| Elements | Raw PA | Pretreated PA | Final Product | Reduction (%) |
|-----------------------------------|--------|---------------|---------------|---------------|
| P ₂ O ₅ (%) | 25.90 | 25.83 | 25.15 | 2.63 |
| SO ₄ ^{2−} (%) | 2.05 | 0.01 | 0.009 | 10.00 |
| Al (%) | 0.50 | 0.43 | 0.001 | 99.77 |
| Fe (%) | 0.28 | 0.27 | 0.001 | 99.63 |
| Ca (%) | 0.23 | 0.25 | 0.010 | 96.00 |
| Mg (%) | 0.39 | 0.41 | 0.003 | 99.27 |
| K (%) | 0.04 | - | 0.005 | 87.50 |
| Na (%) | 0.10 | - | 0.020 | 80.00 |
| Si (%) | 1.02 | - | 0.050 | 95.10 |
| F (%) | 1.57 | 0.10 | 0.050 | 50.00 |
| Cd (ppm) | 15.00 | 16.00 | 0.500 | 96.88 |
| Co (ppm) | 0.79 | 0.79 | 0.002 | 99.75 |
| Cr (ppm) | 240.00 | 215.00 | 0.800 | 99.63 |
| Cu (ppm) | 30.00 | 30.00 | 0.700 | 97.67 |
| Mn (ppm) | 14.00 | 14.00 | 0.070 | 99.50 |
| Mo (ppm) | 4.00 | 4.00 | 0.170 | 95.75 |
| V (ppm) | 208.00 | 187.00 | 0.720 | 99.61 |
| Zn (ppm) | 285.00 | 285.00 | 1.650 | 99.42 |
| As (ppm) | 17.00 | 3.00 | 3.00 | 0.00 |
| REEs (ppm) | 60.00 | 60.00 | 1.00 | 98.33 |

4. Conclusions

A functionalized and conditioned nanofiltration membrane was used to purify industrial PA. Lab-scale pilot tests showed promising results in reducing undesirable impurities. The use of membranes along with pretreatment steps to remove arsenic, fluorides and sulfates allowed the production of food-grade PA. The reductions of the undesirable impurities of Cd, Al and Fe, rather than valuable REEs, were 94.81, 99.30, 99.63 and 96.67%, respectively.

PA purification by functionalized membrane was controlled by the electrostatic repulsion between the membrane and highly charged cation. Moreover, the reduction rate of metals followed the order: trivalent cation > divalent cation > monovalent cation.

Industrial pilot scale tests for PA purification using membrane technology with an outflow of 160 L/h are currently ongoing at Jorf Lasfar fertilizer center. Altogether, the obtained results demonstrate the stability of the membrane within an approved reduction rate of metals slightly higher than the reduction rate at laboratory scale.

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