Journal of Science with Technological Applications

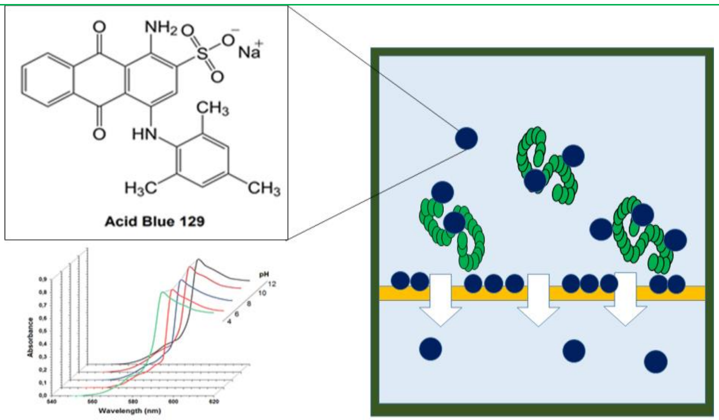
Research Article

Manuel Palencia[1*], **Jina M. Martínez**[2], **Álvaro Arrieta**[3]

[1] *Research Group in Science with Technolgical Applications (GI-CAT). Department of Chemistry, Faculty of Natual and exact Science. Universidad del Valle, Cali – Colombia.*
[2] *Mindtech Research Group (Mindtech-RG), Mindtech s.a.s., Cali - Colombia*
[3] *Department of Biology and Chemistry, Faculty of Education and Sciences, Universidad de Sucre, Sincelejo - Colombia.*

Corresponding author: manuel.palencia@correounivalle.edu.co

Graphical abstract



Removal of Acid Blue 129 dye by Polymer-Enhanced Ultrafiltration (PEUF)

Abstract

There are different techniques which are used for removal of dyes from aqueous effluents. To carry out biochemical and chemical oxidation processes is needed the grouping and concentration of dyes in order to ease the degradative process. The above can be achieved by the use of polymer-enhanced ultrafiltration (PEUF); this is method continuous, environmentally friendly and easily adaptable to features of effluent and nature of dye. Therefore, the objective of this work was to study the use of PEUF for Acid Blue 129 (AB129) removal from aqueous effluents in order to be used as previous stage to its degradation by oxidation processes. PEUF experiments were performed using poly(allyl(triethyl)ammonium chloride) as water-soluble polymer, cellulose membranes and an ultrafiltration unit with dead-end flow under different values of pHs. Also, experiments in absence of polymer were performed. Results shown that two stages can be identified during the retention experiments, a first stage of flocculation followed of a stage

Keywords
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Synthetic dye
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Remoción de Acid Blue 129 mediante Ultrafiltración asistida por Polímeros (PEUF)

Resumen

Hay diferentes técnicas para la remoción de colorantes de efluentes acuosos. Para llevar a cabo procesos de oxidación química y bioquímica es necesario el agrupamiento y concentración de colorantes con el fin de facilitar el proceso de degradación. Lo anterior puede ser alcanzado por el uso de ultrafiltración asistida por polímeros (PEUF); este es un método continuo, ambientalmente amigable y fácil de adaptar a las características del efluente y la naturaleza del colorante. Por lo tanto, el objetivo de este trabajo fue estudiar el uso de PEUF para la remoción de AB129 de efluentes acuosos con el fin de usar esta técnica como una etapa previa a su degradación por procesos oxidativos. Experimentos de PEUF se realizaron usando poli(cloruro de alil(trietil)amonio) como polímero soluble en agua, membranas de celulosa y una unidad de ultrafiltración con flujo de punto final ciego bajo diferentes valores de pH. También, se realizaron experimentos en ausencia de polímero. Los resultados muestran que dos etapas pueden ser identificadas durante los experimentos de retención, una primera etapa de floculación seguida de una etapa de elución, siendo la retención de AB129 promedio en todos los casos del 96 %. En ausencia de polímero, los valores de retención estuvieron entre el 28 y el 45 %, siendo el orden en función del pH 4 ~ 6 > 8 > 10 > 12. Además, se determinó que el modelo de zonas de distribución usado para la diafiltración de iones en PEUF puede ser inapropiado. Los experimentos en ausencia de polímero sugieren que la membrana debería considerarse como un absorbente durante el proceso en lugar de un elemento pasivo de separación.

Palabras claves
Acid Blue 129
Colorante sintético
Ultrafiltración
PEUF
Tratamiento de aguas

Recived: 05-03-2017
Accepted: 12-04-2017
Publishing date: 15 – May – 2017
Revision Code: 20170305-MFMC [Pag. 65-74]

Corresponding author:
manuel.palencia@correounivalle.edu.co

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Abstract

There are different techniques which are used for removal of dyes from aqueous effluents. To carry out biochemical and chemical oxidation processes is needed the grouping and concentration of dyes in order to ease the degradative process. The above can be achieved by the use of polymer-enhanced ultrafiltration (PEUF); this is method continuous, environmentally friendly and easily adaptable to features of effluent and nature of dye. Therefore, the objective of this work was to study the use of PEUF for Acid Blue 129 (AB129) removal from aqueous effluents in order to be used as previous stage to its degradation by oxidation processes. PEUF experiments were performed using poly(allyl(triethyl)ammonium chloride) as water-soluble polymer, cellulose membranes and an ultrafiltration unit with dead-end flow under different values of pHs. Also, experiments in absence of polymer were performed. Results shown that two stages can be identified during the retention experiments, a first stage of flocculation followed of a stage of elution, being the average AB129 retention in all cases 96 %. In absence of polymer, values of retention among 28 and 45 % were obtained, being the observed order as a function of pH: 4 ~ 6 > 8 > 10 > 12. In addition, it was determined that model of distribution zones used for diafiltration of ions in PEUF can be inappropriate. Experiments in absence of polymer suggest that membrane should be considered to act like adsorbent instead to be a passive separation element.

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1. Introduction

Textile industry is a sector plays an important role in world manufacturing industry and is related with the design and production of yarn, cloth, clothing, and their distribution. This industry uses as raw natural or synthetic material and different chemical products; in addition, it uses large volumes of water in wet processing operations, and therefore, generates large amounts of wastewater with enormous dissolved dyestuffs amounts and other products such as dispersing agents, dye bath carriers, salts, emulsifiers, leveling agents and heavy metals; However, the characteristics of textile industry effluents vary and depend on the type of textile manufactured and the chemicals used. Problems associated with textile dyes are not only the color production to the aqueous stream, textile dyes can be carcinogenic, genotoxic and teratogenic substances [1-4].

The nature and origin of dyes are the main criteria used for the general classification of natural and synthetic textile dyes; but also, dyes can be classified with respect to their chemical structure (*e.g.*, azo, anthraquinone (AQN), indigo, triphenylmethane), the method of usage (*e.g.*, direct, reactive, chromic, metal-complexes, disperse, mordant, sulphur, vat, pigments), and/or their chromogen (*e.g.*, donor–acceptor, cyanine, polyenes) [5-7].

Azo and AQN dyes are the two major classes of synthetic dyes and pigments. Together they represent about 90 % of all organic colorants. The exact world production rate of synthetic dyes is not known, however, it is estimated that it is around 10,000 tons/year, of which approximately between 1-2 % and 1-10 % pass, respectively, to the discharge current to the environment at the production stage and during the use. Azo and AQN dyes, or degradation subproducts, can produce high environmental contamination and serious damages to the human health due to its genotoxic activity, by direct chemical interaction (*e.g.*, Azo-type dyes) or by intercalation in the molecule of DNA (*e.g.*, Azo-type dyes), fact that contrasts negatively with its commercial importance [7]. In addition, as dyes are designed to be chemically and photolytically stable, they are highly persistent in natural environments [5-7]. Analysis of over 200 acid dyes indicates that some monoacid and diacid dyes show moderate to high toxicity (that is acute values < 100 mg/L and < 1.0 mg/L) to fish and aquatic organisms [8].

In particular, Acid Blue 129 (AB129) also called 1-amino-4-(2,4,6-trimethylphenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonic acid, is an acidic dye, AQN-type dye, which is widely used for the dyeing of cotton, wool, silk, nylon, paper and leather [2]. By structure-activity study, AB129 has been reported as a highly selective antagonist at the P2Y-receptor of the guinea pig [9-11]. In addition, it associated to possible genotoxic activity and environmental toxicity [8].

On the other hand, polymer enhanced ultrafiltration (PEUF) or liquid-phase polymer-based retention (LPR) is a hybrid method of membrane separation characterized by the use of water-soluble polymers and ultrafiltration membranes. In PEUF, a water-soluble polymer solution and a target specie solution are contacted in the feed-side of an ultrafiltration system; the target species are consequently bonded to the polymer and new macromolecular species are formed and retained by a size exclusion mechanism while the unbounded species, with a diameter smaller than cutoff diameter of the membrane, passes to the permeate stream [12, 13]. An illustration of separation mechanism and main components of the system are shown in the Figure 1.

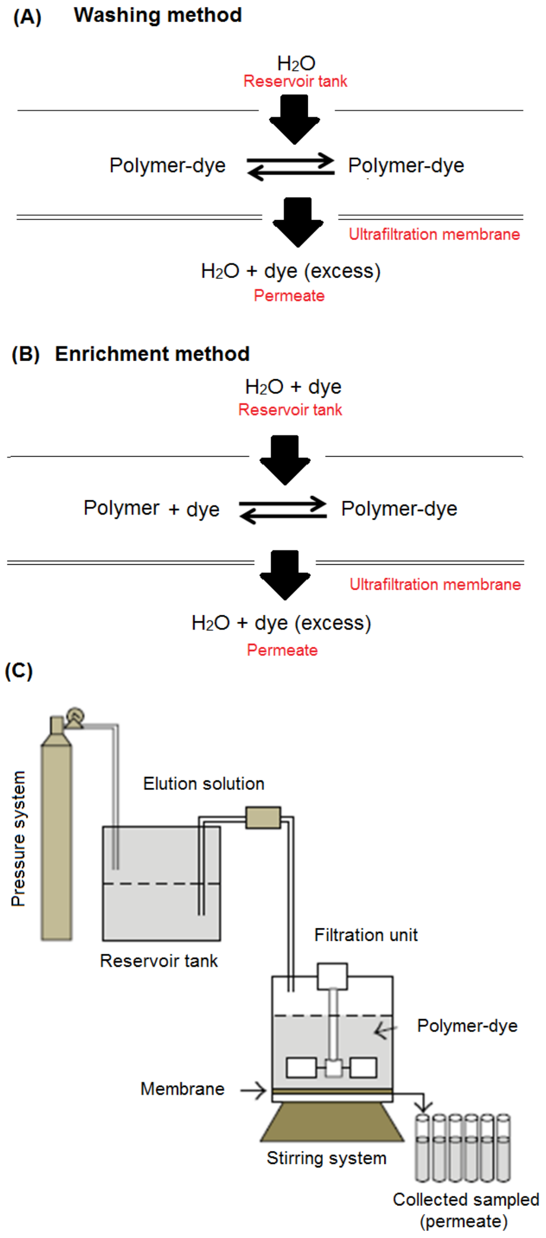

Figure 1. Illustration of PEUF: (A) washing method, (B) enrichment method and (C) components

Usually LPR technique can be applied by two operation methods: (1) the washing method, or elution method, with the ionic strength kept constant or not, which is the most used method to study the retention capacity of a polymer in aqueous solution and (2) the enrichment method, or concentration method, which is a continuous separation process [5]. In the washing method, a certain amount of polymer and target substance solutions, usually metal ion solution, with known concentrations are placed in the feed-side of an ultrafiltration cell and a water stream is passed through the cell [12-15]. For the application of the enrichment method, the polymer solution is placed in a stirred cell and a solution of target species is added continuously from a pressurized reservoir. However, when the bonding groups of the polymer are saturated by their interaction with the substances in the solution, the maximum retention capacity (MRC) of polymer is reached and the process cannot continue [12, 13]. Evidently, the physicochemical properties of the polymer and the polymer-metal complexes must be taken into account and their interaction with the membrane surface is also very relevant.

The PEUF technique can be visualized like cyclic separation process and thus it should be economically more feasible when water soluble polymer can be regenerated and reused. Among regeneration methods of the polymer are the chemical methods (protolysis, transcomplexation, and redox reaction), electrochemical methods, and thermal methods [13]. In this way, new options of regeneration of chelating groups by using new components incorporated to the system can be useful to make the process more feasible [12, 16]. During the retention and separation process by PEUF, the permeate is removed at the same rate, keeping the volume in the feed cell and the polymer concentration constant all the time [13]. Reviews on PEUF technique have been performed by Geckeler *et al*. (1986) [17] and most recently by Rivas *et al.* (2011) [12] and Palencia (2015) [18].

There are different techniques used for removal of dyes from aqueous effluents. Some examples are coagulation, nanofiltration, ozonolysis, oxidation and adsorption [2]. To carry out biochemical and chemical oxidation processes is needed the grouping and concentration of dyes in order to ease the degradative process. The above can be achieved by the use of polymer-enhanced ultrafiltration (PEUF); this is a method continuous, environmentally friendly and easily adaptable to features of effluent and nature of dye. Therefore, the objective of this work was to study the use of PEUF for AB129 removal from aqueous effluents in order to be used as previous stage to its degradation by oxidation processes.

2. Experimental section

2.1. Reagents and filtration unit

AB129 was obtained from Aldrich and used as acidic AQN-type dye model (see Figure 2A). disk-shaped membranes of cellulose (Biomax PBGC; nominal molar mass cutoff of 10 kDa, Amicon Bioseparations-Millipore Co.) were used in all experiments. Experiments were performed in a stirred-cell filtration unit (Millipore, model 8050).

Poly(allyl(triethyl)ammonium chloride) was synthesized from allyl(triethyl)ammonium chloride (Aldrich) and used as water soluble polymer. Synthesis of analogous polymers has been previously published and it is not shown in this paper [19-20]. Components and operation modes of the filtration system have been described in previous publications (see Figure 1C) and include a reservoir tank, filtration cell, magnetic stirring system, ultrafiltration membrane, and pressure system [13].

2.2. Retention experiments and study of fouling

Retention experiments were performed by washing method. In each experiment, 10 mL of a 100 mg/L polymer solution was mixed with 20 ppm AB129 solution at different pH (4, 6, 8, 10 and 12). The effect of pH on absorption maximum for AB129 was analyzed in order to determine the working wavelength. The operating pressure and stirring rate were 300 kPa and 200 rpm, respectively. These operating conditions have been previously established in experiments of ion retention [12, 13, 15]. Permeate fractions with a volume of 20 mL were collected, and the AB129 concentration was measured by atomic absorption spectroscopy (AAS, UnicamSolaarM5).

A blank experiment was performed using the same experimental conditions at the different working pHs. Impact of process on membrane was studied by scattering electron microscopy (SEM) and permeability test.

3. Results and discussion

3.1. UV spectrum of AB129 in function of pH

AB129 structure is shown in the Figure 2A. It can be seen that has groups with nitrogen and sulfur (amine and sulfonate, respectively). In addition, it has two structural units, AQN and 1,3,5-trimethylbenzylamine, which constitute the chromophore core. In the Figure 2B, the UV spectrum of AB129 at different pH are shown. It can be seen that maximum absorption band, at 594 nm, is not affected by pH among 4-12. For wavelengths lower than 594, a small effect of pH lower than 10 on the features of UV spectrum was evidenced.

Differential spectra are shown in the Figure 3 in order to analyze the changes resulting of pH. Thus, it can be seen that absorption band at 585 nm disappears at values of pH 10 and 12.

It has been reported that UV spectra of AQNs show four $\pi \rightarrow \pi^*$ absorption bands in the wavelength range 220–350 nm (*i.e.*, due to $s\pi\rightarrow\pi^*$ electronic transitions) and one $n \rightarrow \pi^*$ absorption band at longer wavelengths, close to 400 nm (*i.e.*, due to $sn\rightarrow\pi^*$ electronic transitions). The position and intensity of the absorption bands are strongly affected by the nature of the auxochromic substituents and environment (polar or apolar solvents). The UV/vis spectra of 1-hydroxy-anthraquinones is determined primarily by their tautomeric or conformational structures (Fain et al. 2006). Intra- and intermolecular hydrogen bonding cause displacement towards longer wavelengths due the formation of a pseudo-ring through hydrogen bond, which increases the length of the conjugated system [21, 22]. Auxochromic substituents have at least one pair of *n* electrons capable of interacting with the π electrons of the ring. This interaction apparently has the effect of stabilizing the π^* state, thereby lowering its energy, and increasing the wavelength of corresponding band. However, if displacement of band overlaps some adjacent band then an increasing in the absorbance is evidenced. Being that it is known that $-NH_2$ have an auxochromic effect on the benzene chromophore, or as it is shown from our data for AQN ring, this is congruent with results that evidence an increasing in the absorbance at 594 nm when system is at pH 10 and 12 (0.81 for pH 10 and 12, whereas for pH 6 and 8 the absorbance values were 0.75 and 0.77, respectively).

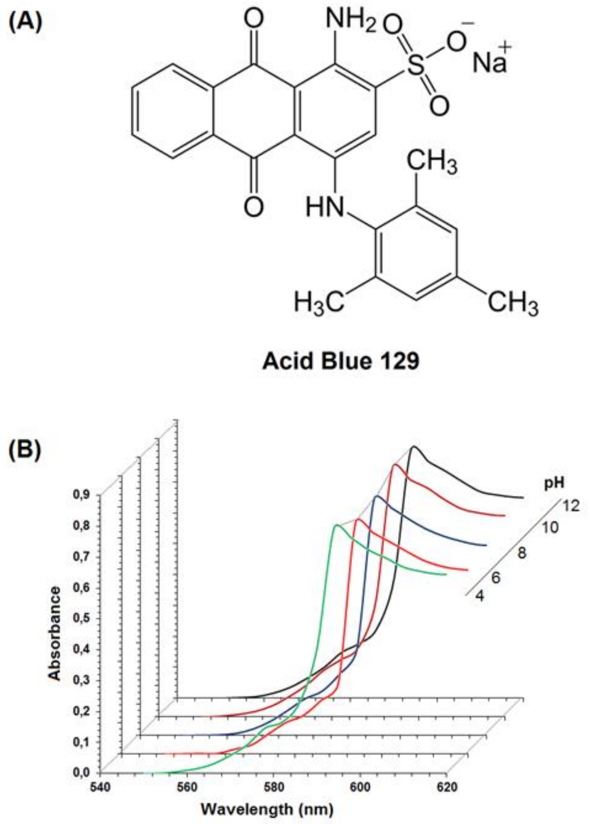

Figure 2. Structure of AB129 (A) and UV spectrum of AB129 at different values of pH (B)

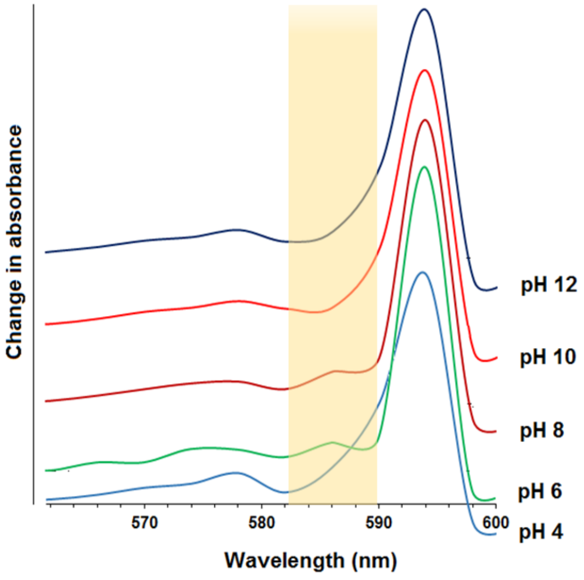

Figure 3. Differential UV spectrum of AB129 at different pH (Yellow mark shows the main change as a result of pH)

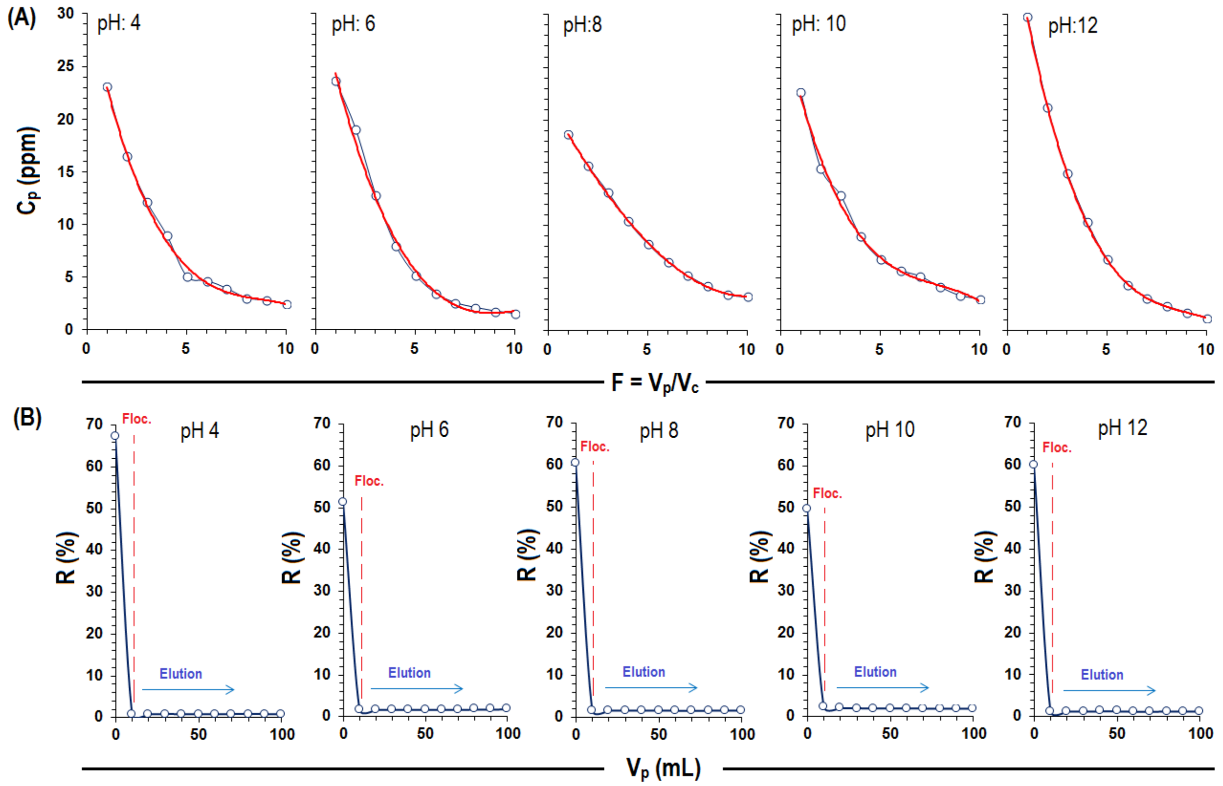

Figure 4. (A) Change of concentration in the permeate in function of filtration factor (*F*). (B) Retention profiles of AB129 (flocculation process “Floc” and Elution process in red and blue, respectively).

3.2. Retention experiments

The behavior of AB129 concentration in function of permeate volume for all working pHs is shown in the Figure 4A. It can be seen that the AB129 concentrations change with the increase of filtration factor (*F*). The observed changes show the same behavior than permeate behavior during elution experiments by PEUF (progressive decrease of concentration with the increase of *F*). However, in the inside of the ultrafiltration cell, the behavior of AB129 concentrations is different to that observed during diafiltration experiments (instead of a progressive decrease of concentration with the increase of *F* was observed an Abrupt drop in dye concentration at small *F* values). The above suggests that the process during retention experiments is not completely analogous to removal of ions by PEUF. The average AB129 retention were in all cases among 95-97 %. However, two stages can be identified during the retention experiments, a first stage of flocculation followed of a stage of elution. In addition, the 90 % of retention contribution can be explained by the first stage (see Figure 4B). Results evidence that there is no effect of pH (from 4 to 12).

3.3. Study of fouling by adsorption of AB129

Fouling is the main problem in the process of the membrane because limits the continuity of process and changes the permeability features of the membrane. Thus, during the filtration of AB129, concentration changes with time analogously to diafiltration of metal ions. This observation was different when experiments were performed in the presence of polymer. It can be seen that behavior of retention (*R*) as a function of permeate volume (V_p) in the Figure 5A. Values of retention among 28 and 45 % were obtained, being the observed order as a function of pH the following: 4 ~ 6 > 8 > 10 > 12. The efficiency of dye adsorption is dependent on the solution pH, because variation in pH leads to the variation in the degree of ionization of the adsorptive molecule.

Retention of AB129 by the membrane evidence a strong interaction between membrane surface and dye, in addition, indicate that the process described in the Figure 4B is the result of three process which act simultaneously: flocculation by the polymer interaction, adsorption on the membrane surface and diafiltration (or elution) from solution through the ultrafiltration membrane. In the elution, different residence times were observed, but this can be a result of different concentrations in the cell at the initial time.

In the model of distribution zones, when AB129 solution is introduced in the ultrafiltration unit, this is distributed in different zones (bulk solution, membrane and permeate), being the saturation rate of membrane very fast through irreversible process and, in consequence, the changes in the bulk and in the permeate can be described by

$$\frac{C_p}{C_0} = \frac{\alpha\theta}{\alpha\theta + F} \qquad (1)$$

where $\theta = 1 + k_m\, w_m\, V_c^{-1} + k_{pol} V_{pol} V_c^{-1}$. C_p, C_0, α and F are the permeate concentration, initial concentration of AB129 in the inside of ultrafiltration cell, hydrodynamic parameter and filtration factor ($F = V_p/V_c$ being V_p and V_c the volume of permeate and in the inside of the ultrafiltration cell), respectively. In addition, k_m and k_{pol} are the interaction parameters dye-membrane and dye-polymer, respectively. If diafiltration experiment is performed in absence of polymer, then $k_{pol} = 0$. Mass of membrane is denoted as w_m [15, 18]. However, the use of model of distribution zones evidence that passive elution is not occurring. Model predicts a linear plot for C_p versus $1/V_p$ and this is not seen (see Figure 5B). This non-linear tendency can be explained by a strong interaction with the membrane through the time.

Results of permeability test are shown in the Figure 6. It can be seen that there is not significant difference between hydraulic permeability at different pH. This result suggests that pore blocking pore is not produce, including cake filtration, internal, standard and intermediate blocking [15]. Also, it is suggested that retention is mainly produced on the surface and could be an adsorption process with low capacity of fouling formation.

Similar results have been reported by Majewska-Majewska-Nowak *et al*., (2006). They found that mechanism governing the SDS separation from a solution containing anionic dyes can be explained by the adsorption of surfactant monomers and dye particles in the membrane pore interior [19-20]. However, clearly the presence of surfactant is a differentiator factor respect to our experiments.

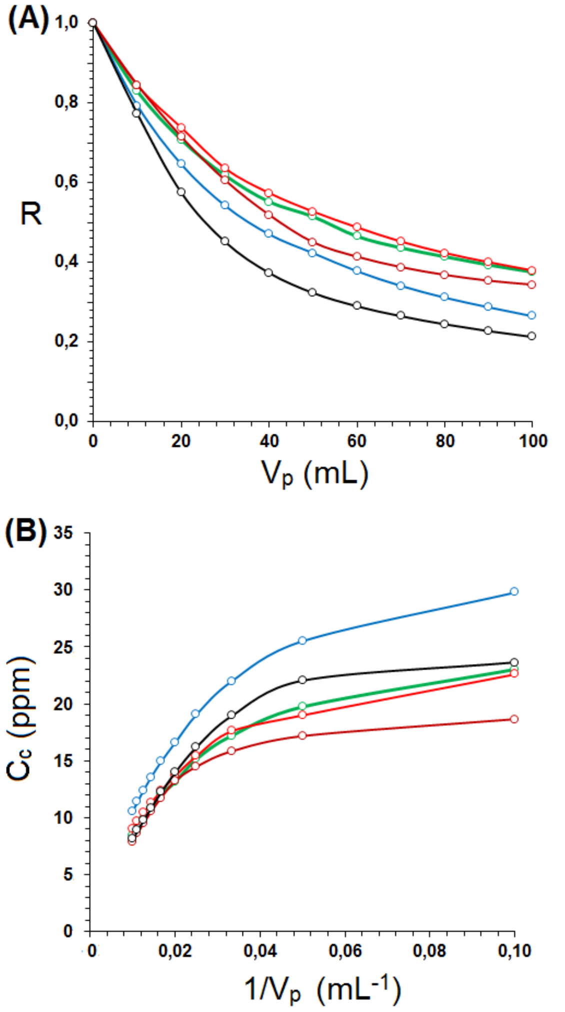

Figure 5. A) AB129 retention (*R*) as a function of permeate volume (V_p) (Top-down pH: 4, 6, 8, 10 and 12) and B) changes of cell concentration as a function of $1/V_p$.

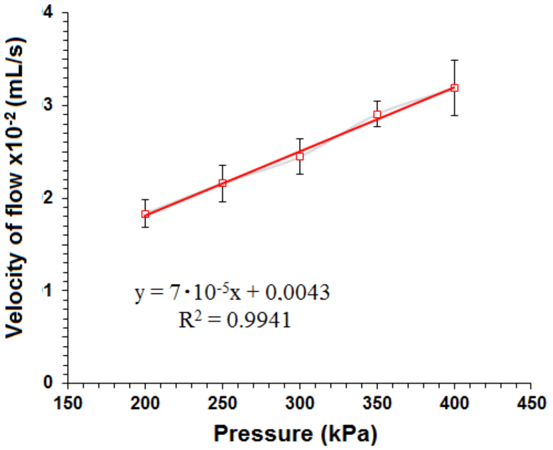

Figure 6. Velocity of flow as a function of pressure after to diafiltration experiments at different pHs.

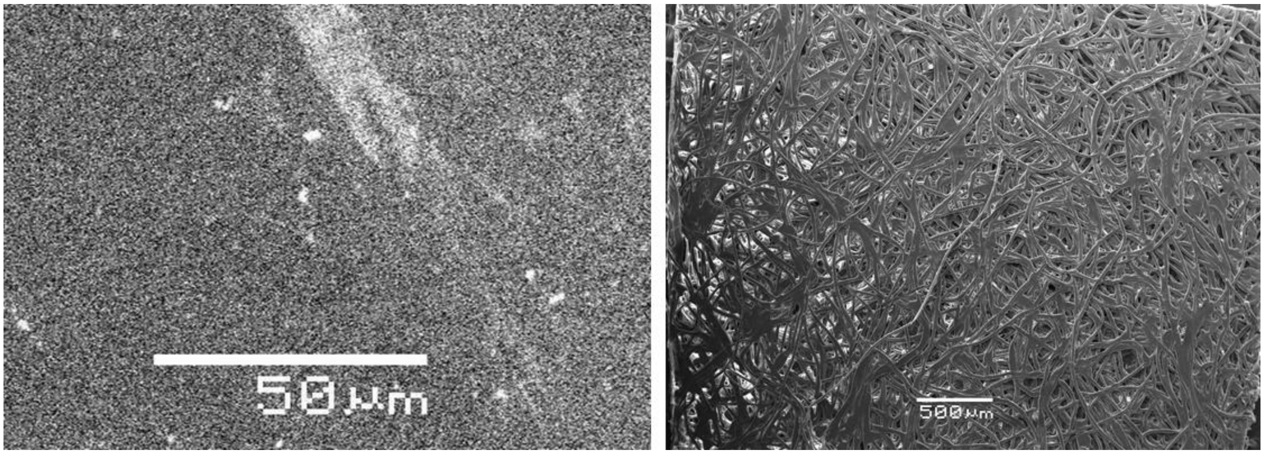

Figure 7. SEM images of membrane after to diafiltration experiments: active layer (left) and support (right).

Finally, experiments in absence of polymer suggest that membrane should be considered to act like adsorbent instead to be a passive element to produce the filtration.

Images of SEM are shown in the Figure 7. It can be seen that membrane is the asymmetrical structure with active layer constituted by cellulose with a very small pore diameter compared with support layer of polypropylene. However, significant changes were not observed. This is congruent with the results obtained by permeability test.

4. Conclusions

Results shown that two stages can be identified during the retention experiments, a first stage of flocculation followed of a stage of elution, being the average AB129 retention in all cases 96 %. In absence of polymer, values of retention among 28 and 45 % were obtained, being the observed order as a function of pH: 4 ~ 6 > 8 > 10 > 12. In addition, it was determined that model of distribution zones used for diafiltration of ions in PEUF can be inappropriate. Experiments in absence of polymer suggest that membrane should be considered to act like adsorbent instead to be a passive separation element.

Acknowledgment

Authors thanks to Universidad del Valle and Mindtech s.a.s. to funds for the performing of this research. Also, to the Program for Scientific Knowledge Diffusion of Mindtech s.a.s. (PSKD 2016-2020) for funds associated to the publication costs.

Conflict of interests

The authors declare no conflict of interest.

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