Investigation of dye removal from aqueous solutions by Preyssler assisted-ultrafiltration: UV-visible and photoluminescence study

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Abstract
The aim of this work is to study the interaction between preyssler type polyoxometalate K14 [NaP3W29Mo1O110] (P5M30) and crystal violet (CV) dye using UV–visible (UV–vis) and photoluminescence (PL) spectroscopy. The complexation-ultrafiltration method is also studied in order to retain CV from aqueous solution using for the first time the P5M30 as complexing agent. The addition of P5M30 polyanions to the CV solution leads to the formation of metachromatic complex. They revealed by the blue shift of the absorption maxima of the dye. This variation is attributed to the formation of dye h-aggregates. The emission spectra show a progressively decrease in the intensity band as function of P5M30 concentration increase which indicate that there is an interaction between dye and P5M30. The Preyssler assisted-ultrafiltration reveals that the CV retention was significantly enhanced from 10% to 98% following the addition of P5M30. The effect of chemical additives such as P5M30, NaCl and CTAB concentrations, the pH and the applied pressure on the dye retention and permeate flux have been also investigated.

1. Introduction
Large varieties of industrial effluents contain great quantities of synthetic organic dyes. The release of these compounds in the environment leads to greater pollution, thus a serious risk to health [1, 2]. Therefore, industries have been obliged to get new alternatives for the treatment of their effluents before its discard into the environment [3, 4]. In this circumstance, the enforcement of membrane technology becomes a potential way for the treatment of polluted water [5, 6]. Generally, the nanofiltration and the ultrafiltration membranes have shown great efficiency for the removal of ionic pollutants in both performances and economic terms [7]. The separation process of ionic pollutants by the membrane is mainly according to the size as well as to the electrical mechanisms [8]. The large pores of UF membranes lead to significant permeation fluxes while weak ion retention. On the other hand, the sealed pores of NF membranes induce high rejections, but accompanied by low permeate fluxes. Therefore, several studies were focused to enhance rejection performances without disrupting the permeate flux [9, 10]. Among the promising solutions, there are the uses of polymers as membrane modifiers. Hence, polymers can modify the physicochemical properties of the membrane so as to raise its retention performances [11–13]. Furthermore, hybrid process consisting with the use of additives in the feed solution can also be a promising way to enhanced pollutant retention. The increase of the effective size of the contaminant ions by complexation leads to great retention enhancement. This method is namely polymer-enhanced ultrafiltration (PEUF) when polymer was used as complexing agents while it’s called micellar enhanced ultrafiltration (MEUF) when surfactants present the additive. Generally, PEUF is mostly used to eliminate dye molecules by adding synthetic polymer such as polyacrylic acid (PAA) and polyammonium acrylate (PANH4+) [14, 15] or polyethyleneimine (PEI) [16]. Likewise, MEUF is also shown high efficiency to eliminate dyes using cetylpyridinium chloride (CPC) [17] and sodium dodecylsulfate (SDS) [18].

Polyoxometalates, are discrete molecular structures composed of cationic metals bridged by oxide anions, typically named clusters. They have been used widely to solve various environmental problems thanks to their
miscellaneous chemical properties such as Brønsted acidity, redox and photochemical reduction properties, magnetism, and considerable sizes [19–22]. Among the large POMs clusters, we mention the Preyssler anion with general formula [NaP5M30O110]14−. Their structure consists of a cyclic assembly of five PW6O22 units; each derived from the Keggin anion, [PW12O40]3−, by removal of two sets of three corner-shared WO6 octahedra.

Recently preyssler anion shows its good properties in catalysis and material science [23, 24]. In addition, it was recently used for the treatment of radioactive waste [25–27]. Several studies have been described by Hafiane and coworkers to eliminate different dyes from aqueous solution. For instance, they described the removal of CV using Coffee waste as potential adsorbent [28], removal of methyl orange using cationic surfactants modified coffee waste [29], removal of methylene blue by polyelectrolytes assisted ultrafiltration [14], removal of Safranin T, methylene blue and Direct Blue 71 using micellar enhanced ultrafiltration [30–32]. As a continuation of this sense, we report for the first time the use of Preyssler anion, H14[NaP5W29MoO110], as complexing agents to enhanced ultrafiltration of crystal violet (CV) dye. Especially, the effect of transmembrane pressure, P5M30 anion concentration, salt addition and solution pH value on CV removal performances were presented and discussed.

2. Experimental section

2.1. Chemicals

The cationic phenothiazine dye, Crystal violet (molecular weight: 407.99 g mol−1, λmax = 590 nm (figure 1)), Sodium chloride, Hydrochloric acid, sodium hydroxide, sodium tungstate, sodium molybdate, disodium hydrogen phosphate, acetic acid, potassium chloride were purchased from Fluka. All the chemicals were used without further treatment. The water used for all prepared solutions is the distilled water.

2.2. Synthesis of P5M30

The structure of prepared Preyssler polyanions is shown in figure 1. The synthesis of P5M30 was described as the same protocol mentioned in the literature [23]. Briefly, appropriate amount of sodium tungstate and sodium molybdate were dissolved in water (35 ml). The solution was mixed for 30 min at 60 °C. Then phosphoric acid 85% was added. In the next step, the mixture was sealed into a Teflon bottle at 120 °C for 18 h. Then, a solution of potassium chloride was added into the above mixture to form the precipitate. The obtained product was dried and recrystallized twice to obtain the potassium salt of P5M30.

2.3. Characterization techniques

IR spectrum was recorded by a Perkin Elmer (FTIR 2000) spectrometer in the range of 4000–400 cm−1 using KBr pellets. The UV–visible spectrum was recorded with a Perkin-Elmer Lambda 25 spectrophotometer using a matched pair of glass cuvettes with 1 cm optical lengths. The fluorescence spectra were performed by Cary Eclipse Fluorescence Spectrophotometer (Agilent technologies) in the range of 400–800 nm. The solution pH was controlled and corrected using pH meter (AZ86505, AZ Instrument).

2.4. Procedure

For UV–vis and PL measurement, all solutions were prepared by adding aliquots of P5M30 solution (ranging from 0 to 7.5 ml in order to vary the ratio of the P5M30 to dye) to a fixed volume of 2.5 ml of the CV solution. The final volume of the solution was adjusted to 25 ml by adding distilled water, making the final dye concentration in the
solution at $2.10^{-3}$ mol·l$^{-1}$. The mixture of CV dye and P$_5$M$_{30}$ solutions were stirred evenly for 30 min prior recording absorption and emission measurement.

The absorbance measured at $A_m$ present the absorbance of monomeric band (590 nm) while $A_M$ refers to the absorbance of metachromatic band (530 nm). $A_m/A_M$ ratio was plotted against the Preyssler/dye ratio $[P]/[D]$ to investigate the stochiometry of the complex using the ratio method.

UF experiments were performed in 50 ml Amicon 8050 with an effective membrane area of 15.54 cm$^2$. Regenerated cellulose ultrafiltration membrane with a MWCO of 3 kDa was used. The schematic diagram and the process of ultrafiltration are shown in figure 2. The filtration tests were performed at a total pressure of 2 bars except for pressure effect. Furthermore, Constant stirring in the cell was maintained at a speed of 300 rpm and the pH of solutions was adjusted by adding hydrochloric acid or sodium hydroxide solutions (0.1 M). After filtration, fractions of 5 ml were collected in permeate reservoir in order to measure the final dye concentration using UV-Vis spectroscopy. After each manipulation, the membrane was immediately washed with distilled water to assess its cleanness.

The permeate flux and the dye retention are the two parameters that describe the filtration performance. So flux was calculated analytically from the correlation of total volume of permeate collected versus time from the following equation (1):

$$J_v = \frac{V}{\Delta t \times S},$$

Where $V$ (L); the volume of the permeate, $S$ (m$^2$); the effective membrane area and $\Delta t$ (h); the permeation time.

The retention coefficient (%) of the membrane is defined by the following equation (2):

$$R\% = \left(1 - \frac{C_p}{C_o}\right) \times 100,$$

Where $C_o$ is the initial concentration of the dye in the feed and $C_p$ is the dye concentration in permeate solution.

3. Results and discussion

3.1. Fourier transform infrared spectroscopy (FTIR)

In order to get better insight into the chemical bonds in our product, vibrational FTIR measurement was used. The IR spectrum of P$_5$M$_{30}$ anion powder is shown in figure 3. As can be seen, a broad band appeared at 3572 and 1615 cm$^{-1}$ is attributed to the vibration of O–H of water molecules. The absorbance band located at $\sim$929 cm$^{-1}$ is related to the vibration of (M–Ob–M). We have also observed the absorption band at 593 cm$^{-1}$ related to the asymmetric vibration of (O–P–O) link. The band located in the region of 1087 cm$^{-1}$, is attributed to P–Oa [33].

3.2. UV-visible spectroscopy

Figure 4 presents the UV–visible spectrum of P$_5$M$_{30}$ registered between 200 and 800 wavelength. From this figure, a band appeared at 212 nm and 284 nm are attributed to the metal charge transfer (LMCT). We note that the band at 212 nm is due to the transfer charge from O$_t$ (terminal oxygen) to metal. However, the band around 284 nm is due to the charge transfer of the bridging oxygen Ob and Oc to metal [34].
4. UV–visible and photoluminescence investigation

4.1. P5M30 concentration effect

Figure 5 illustrates the UV–vis spectra of the CV dye in presence of P5M30. Generally, in aqueous solution, CV exhibits an intense band at 590 nm and two shoulders at 250 nm and 303 nm. The degradation rate of the dye was investigated at the most intense wavelength at 590 nm. As seen in Figure 5, the addition of low P5M30 concentration to the CV solution (P/D = 0.1) causes the decrease of the monomeric absorption band at 590 nm. Furthermore, at P/D ratio ranging from 0.5 to 10, a new band around 530 nm (blue shift) was appeared gradually with the disappearance of the monomeric band. According to Schiller et al [35], this behavior may due to the formation of higher aggregates of dye molecules like the dimer (CV)2 with the increase of P5M30 polyanions. Indeed, the blue shift corresponds to the formation of metachromatic complex type H-aggregates. The metachromasia phenomenon is the results of the charge transfer between cationic and anionic ions mixed in aqueous solution [36]. Near the P5M30 polyanions the dye self-aggregates with a parallel organization face to face through the aromatic-aromatic interactions [37–39]. In addition, the spectra show the absence of isosbestic point; this indicates that the CV confronts more than two environments over the studied concentration range. In accordance with Schiller et al [35], this result can be explained by the equilibriums presented in the following reactions:

\[
CV + P5M30 \rightleftharpoons [CV - P5M30] \quad \text{(a)}
\]

\[
CV + [CV - P5M30] \rightleftharpoons [(CV)2 - P5M30] \quad \text{(b)}
\]

Further, the polyoxometalates of preyssler have generally three chemically active sites: the terminal oxygen (M=O₁), the bridging ones (M–O–M) and particularly the internal oxygens linked to the heteroatom (P–O–M)
which are accessible and even these are the most basic ones [39]. These oxygen acts as inorganic building construction linkers [40]. Thus, these three sites were interacted with CV$^+$ dye (figure 6).

In addition, the stoichiometry of the complexes was determined by UV-vis spectroscopy using the mole ratio method [41]; the absorption spectrum of CV dye exhibits a maximum absorbance value at 590 nm ($A_{390}$). As P$_5$M$_30$ was added to CV solutions, the band at 590 nm shifted to shorter wavelengths and a new band was formed around 530 nm ($A_{530}$). In the molar ratio method, the maximum absorption values of the complex solution ($A_{m}/A_{390}$) with different concentrations are plotted versus the molar ratio of P$_5$M$_30$ to dye (P/D) (figure 5). The obtained molar ratio curve of CV/P$_5$M$_30$ complex would appear to extrapolate to a maximum coordination number of n = 1. The P$_5$M$_30$/CV complex stoichiometry was 1:1, and this result likely evince that each P$_5$M$_30$ anionic site was associated with one cationic dye [14, 42].

On the other hand, in the following section the interaction between P$_5$M$_30$ and CV dye was studied for the first time by fluorescence spectroscopy. Figure 7 presents the PL spectra of CV in presence of P$_5$M$_30$ at various P/D ratios. It can be seen that at P/D = 0, the spectrum show two emission bands: a low band around 385 nm appeared in UV region wavelength and an intense band appeared in visible region (682 nm). However, in presence of P$_5$M$_30$ the fluorescence intensity of CV decreases regularly with the increase of P$_5$M$_30$, keeping the same emission wavelength. This behavior may be explained as follows: the negative sites increases as function of P$_5$M$_30$ concentration increases, thus more dye was interacted with these sites. As a consequence, a decrease in the emission band intensity was observed. This behavior is related to the Fluorescence quenching phenomena [43]. Generally, the fluorescence quenching is the decrease of the quantum yield of fluorescence from a fluorophore.

![Figure 5. Absorption spectra of CV at different P/D ratios and the Stoichiometry of CV/P$_5$M$_30$ complex.](image5.png)

![Figure 6. Mechanism of CV dye interaction with P$_5$M$_30$ anion.](image6.png)
induced by a variety of molecular interactions with quencher molecule (P5M30). Indeed, an explanation was given by Xu et al 2009 [43] indicating that the fluorescence intensity of BSA decreases regularly with the increase of CV concentration. Therefore, the CV could interact with BSA and quench its intrinsic fluorescence. Moreover, the fluorescence intensity is related to the concentration of the quencher. In addition, the fluorescence quenching phenomena are not due to one single mechanism but depends on others behaviors [44, 45]:

- Fluorescence quenching can be dynamic, resulting from collisional encounters between the fluorophore and quencher,
- Or static, resulting from the formation of a ground state complex between the fluorophore and quencher.

It is worth to be noticed that, fluorophores can be quenched by both collision and complex formation with the same quencher.

4.2. Ionic strength effect
The effect of NaCl salt on the CV and P5M30 interaction was studied at fixed P/D molar ratio of 4. The concentration of NaCl was ranging from $10^{-6}$ mol.L$^{-1}$ to $10^{-1}$ mol.L$^{-1}$. Figure 8 shows that the addition of NaCl to the solution leads to a slight decrease of the metachromatic band intensity with keeping the same wavelength position at 530 nm. Also, the visible spectrum demonstrates that the complex formed between CV and P5M30 anion is weakly sensitive to salt addition. This behavior indicates the stability of the P5M30/CV complex. The presence of the ionic strength slightly conceals out the electrostatic interactions. The impact of salt on self association of dye in aqueous medium was previously investigated. Unlike our obtained results, Ben Mahmoud et al [37], Ben Fradj et al [46] and Xiang et al [47] have been found that the salt addition causes the disappearance of metachromatic complex bands.
The emission spectra of CV \((P/D = 4)\) at various NaCl concentrations are depicted in figure 9. It's shown that the emission band intensity increases regularly as a function of the salt concentration increase but the band position was kept shiftless. This result was probably due to the competition between \(\text{Na}^+\) and \(\text{CV}^+\) in the same negative sites onto \(\text{P}5\text{M}30\) anion. Thus, the release of the dye in the solution leads to the increase in the fluorescence intensity.

4.3. pH effect
Figure 10 illustrates the pH effect on the CV absorption spectrum in presence of \(\text{P}5\text{M}30\) at \(P/D = 4\). It seems from this figure that at pH ranging from 2 to 4, the absorption intensity of the metachromatic band was obviously decreased keeping the same wavelength position. Indeed, this behavior may due to the disruption of the \(\text{P}5\text{M}30/\text{CV}\) complex by the presence of excess \(\text{H}^+\) ion in the solution. Furthermore, at pH \(\geq 6\), the metachromatic absorption band keeps the same position and the same intensity. Thus, it is clear that the alkaline medium is more favorable for the dye aggregation [46].

On the other hand, figure 11 presents the fluorescence spectra of CV \((P/d = 4)\) at different pH value. It is clearly shown from this figure that the emission intensity of CV was increased especially at pH = 2. This can be explained by the occupation of the negative sites on the surface of \(\text{P}5\text{M}30\) with \(\text{H}^+\) proton. So, the discharge of free dye molecules into the solution leads to the enhancement of the emission intensity. Otherwise, when the pH value was ranging from 4 to 10, the emission intensity was decreased. This proves that the complexation between the CV molecules and the \(\text{P}5\text{M}30\) anion is favorable in this pH range.

5. Ultrafiltration study

5.1. Dye retention by the membrane
In this paragraph, we attempt to study the effect of pressure on the CV retention and the permeate flux. The CV concentration chosen to study the membrane’s ability to retain dye was \(10^{-4}\) mol.l\(^{-1}\).
From figure 12 (red curve), it can be seen that at transmembrane pressure of 1.5 bars, the spectrum presents a maximum CV retention rate of 15%. Afterwards, the retention was decreased as a function of pressure increase and only 8% of dye was retained at 3 bars pressure. This finding may due to an increase in concentration polarization effect with the increase of the pressure \[48\]. Additionally, the membrane could not efficiently reject dye molecules due to the large pore size (MWcut off is 3 kDa).

Moreover, as shown in figure 12 (blue curve), the permeate flux increases with increasing pressure. Indeed, the increase in permeate flux has generated a more intensive transport of dye molecules to the membrane surface and leads to the observed decrease in dye retention \[17\].

5.2. Preyssler anion effect

The effect of adding P5M30 on the dye retention as well on the permeate flux is presented in figure 13. In fact, an aqueous solution of the P5M30 anion was added to a fixed dye concentration of \(10^{-4}\) mol.l\(^{-1}\) at different P/D molar ratios ranging from 1:1 to 1:10. After stirring the mixture for 30 min, the solution was filtered through the ultrafiltration membrane at operating pressure of 2 bars. Indeed, as can be seen from figure 13 (red curve), the increase of the P5M30 concentration was clearly enhanced the dye complexation process. At P5M30 concentration of \(10^{-4}\) mol.l\(^{-1}\) and \(2 \times 10^{-4}\) mol.l\(^{-1}\), the CV retention rate was in the order of 53% and 79% respectively. However, when the P5M30 concentration was ranging from \(4 \times 10^{-4}\) mol.l\(^{-1}\) to \(10^{-3}\) mol.l\(^{-1}\), the dye retention plateaued at approximately 97%. This means that, the CV dye molecules were interacted with the P5M30 negatives sites. As a result, the CV/P5M30 complex begins to agglomerate forming larger particles near the membrane surface that can be retained by the membrane \[14, 49\]. The maximum dye rejection was achieved at P/D ratio of 4. By comparing with literature, similar behavior is observed as an example, Hammami et al \[50\] used this method to eliminate neodymium from aqueous solution by using poly (sodium 4-styrenesulfonate) and polyethylene glycol and showed excellent retention performance. In addition Huang et al used Micellar enhanced ultrafiltration to remove methylene blue by using sodium dodecylsulfate (SDS) as complexing agents and found a great enhancement in retention rate \[18\].

The permeate flux versus P5M30 concentration is represented in figure 13 (blue curve). It is worth to note that permeation flux obtained was decreased from 128 to 30 l.h\(^{-1}\).m\(^{-2}\) when the P5M30 concentration was...
ranging from $10^{-4}$ to $10^{-3}$ mol.l$^{-1}$. This behavior can be attributed to the increase of viscosity of the feed solution induced by the P5M30/CV complex formation.

In order to confirm that the improvement of the ultrafiltration membrane performance is mainly induced by the interaction between the dye and the P5M30, the UV-Visible spectra of CV alone and in presence of P5M30 ($P/D = 4$) before and after ultrafiltration were done (figure 14). Initially, the UV-visible spectrum of CV in absence of P5M30 presents an intense band located at 590 nm (figure 14 black curve). Thereafter, in presence of P5M30 at $p/d = 4$, figure 14 (blue curve) shows that the absorption band intensity of the CV is dramatically decreased. The result may due to the P5M30/CV complex formation. Furthermore, figure 14 (red curve) presents the UV-visible spectrum of the CV dye in the permeate solution. It seems that the absorption band intensity has become very low (tends to zero). This behavior can be explained by the fact that the CV is totally retained by the membrane.

5.3. Ionic strength effect

Figure 15 illustrates the effect of NaCl on the CV retention and on the permeate flux. The NaCl salt concentration was varied from $10^{-6}$ mol. l$^{-1}$ to 1 mol. l$^{-1}$. CV and P5M30 ratio was fixed at $p/d = 4$. The pressure was maintained at 2 bars.

From this figure (red curve), it can be seen that the retention decrease from 96% to 94% as a function of the increase of salt from $10^{-6}$ mol. l$^{-1}$ to 1 mol. l$^{-1}$. This result is due to a competition between the Na$^+$ ion and the CV$^+$ molecules in the same negative sites onto the P5M30 surface.

Thus, CV$^+$ will be free in the solution and can be easily passed through the pores of the membrane, resulting in the decreased of dye retention. Unlike Mondal et al [15], this study reveals that the addition of competitive ions in complex solution has no significant effect on the retention of dye which remains high.

The permeate flux is represented in figure 15 (blue curve). It is clearly shown that the flux tends to decrease with the increase of the salt concentration. The flux was varied from $38.7$ l.h$^{-1}$.m$^{-2}$ to $30$ l.h$^{-1}$.m$^{-2}$. This trend can be explained by the formation of a deposited layer over the membrane surface caused by the complex
aggregation. This causes the increase of the resistance against the solvent flux. Comparing with literature, same observations were found by Fradj et al and Ouni et al [14, 51].

5.4. pH effect

The effect of pH solution on the dye retention and on the permeate flux is shown in figure 16. The studied pH value was ranging from 2 to 12. It can be seen from figure 16 (red curve) that the retention of dye was in order of 88% for pH = 2 to pH = 8. However, at pH ≥ 8, the retention of dye reaches a maximum value of 99%. So, this behavior can be related to the stability of the formed CV/P₅M₃₀ complex. Indeed, at low pH value, the affinity of P₅M₃₀ anion towards the dye is weakened due to the presence of positive charges H⁺ and so the complex stability was disturbed. As a result, the CV molecules are released into the aqueous solution and pass through the pores of the membrane, resulting in decreased retention. As pH increases, the affinity and stability of the complexes increases and consequently there is maximum dye retention (99%). These results are in good agreement with other studies which have shown maximum retention at basic medium [14, 15, 52].

On the other hand, the variation of the flux as function of pH value is illustrated in figure 16 (blue curve). From this figure, it can be observed that the flux decrease when the pH value increase. Thus, this characteristic flux variation was due essentially to the adsorption of complexes formed between the dye and the P₅M₃₀ on the surface of the membrane that inhibits the passage of solvent. Similar results were reported by Ouni et al [50].

6. Conclusion

In summary, the optical properties of CV dye in presence of preyssler anion have been reported. The UV-Vis investigation reveals the formation of metachromatic absorption band following the addition of P₅M₃₀. This behavior was due to the formation of complex aggregation (P₅M₃₀/CV). The photoluminescence results show that the PL band intensity decreases gradually by increasing the P₅M₃₀ concentration without emission.
wavelength shift. It is also noteworthy that the observed trend is related to the interaction of CV molecules by P5M30 actives sites that caused quenching of his fluorescence.

The preyssler assisted ultrafiltration method has been performed for the first time to remove the CV dye. It was shown that the elimination of dye was significantly improved in presence of P5M30 and reached 99% at pH 6. Unlike the results found in the literature, this work reveals that the addition of salt and the variation of pH solution have no significant impact on the retention rate, which remains significant.

The results point out the preyssler anion as good new candidate used to improve membrane separation performance. It can be argued that the preyssler assisted ultrafiltration present a promising way to remove several pollutants in the future.

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