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Uses of Some Nano-Sized Organic Wastes to Treat Industrial Wastewater

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Abstract

Industrial progress led to environmental pollution and one of this pollution is heavy metals which accumulate in water bodies, and this causes a serious health hazard to all living organisms. Chromium is one of the heavy metals that are used in various industries. It is present in wastewater in two toxic forms chromium (III) and chromium (VI). Agricultural byproducts as green tea spoil (GTn) and pomegranate peels (PPn) are used for metal adsorption. Its particle size ranged from 12 to 43 nm investigated by TEM. It is found that, the adsorption of Cr (VI) ions dependent on different parameters such as pH, contact time, adsorbent dose, and particle size. The results showed that the remova1 efficiency of Cr (VI) ions was 99.98 and 98.95% for both GTn and PPn, at pH 7, 210 min contact time, and 1g/100ml dose. The utilization of these sources of agricultural byproducts would solve some disposal problems as well as techniques in using cheaper materials for the adsorption of heavy metals in aqueous wastewater due to its abundant available, biodegradable, and reuse material.

Key words: Green tea**,** pomegranate peels**,** nanoparticles**,** wastewater, removal chromium ion

1. Introduction

Industrial progress led to environmental pollution and one of the considerable contaminants that affect water resources is the heavy metals accumulation in water bodies. It is highly toxic at both low and high concentrations in soil and water causing a serious health threat to all living organisms [1]. One of the main global environmental problems is water pollution [2]. The organizations enlarge limitations and put laws to the industrial waste treatment, leading to toxic materials do not exceed the acceptable limits. Therefore, attention has been given to industrial waste containing heavy metals, organic dyes, and contaminants [3].

Chromium is used in many industries, including textiles, paints, fungicides, leather tanning, paper, pigments, fuels and photographic materials [4]. Chromium is present in industrial wastewater in two toxic forms: trivalent Cr (III) and hexavalent Cr (VI). Chromium (III) compounds are significantly less toxic than chromium (VI) compounds. Long term exposure to trivalent chromium causes allergic skin reactions and cancer. Chromium (VI) causes skin infections and chest diseases such as lung cancer and gastroenteritis, 71mg/Kg is the oral human lethal dose for Cr (VI), according to the Occupational Safety and Health Association (OSHA) [5], while 0.05 mg/L is the maximum acceptable level in drinking water according to [6], and the maximum limit of disposal 0.1 mg/l Cr (VI) to inland surface waters.

Metal finishing industries and tanneries are the major sources of chromium contamination. As a result of industrial activity, more than 70,000 tons of chromium pollutants are discharged into the environment annually [7].

There are several chemical and physical methods used to remove chromium from aqueous solutions such as; oxidation, reduction, precipitation, filtration, ion exchange, electrochemical treatment, coagulation/flocculation, irradiation method, photochemical, fentons reagents and biological treatment method [8]. However, all the above methods have disadvantages such as high reagent or energy requirements, incomplete metal removal, and are very expensive to control at low concentrations in the wastewaters [9]. However, the adsorption is the proper method due to its flexibility, simplicity of design, low cost, and high efficiency [10, 11]. Adsorption is an effective method for heavy metals remediation. Some activated materials are effective for this task. But, it is very expensive. Therefore,

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many studies have begun to search for alternative natural adsorbent materials as plants that can be used to adsorbate heavy metals from water systems and soil

Recent methods for Cr ions removal are based on the use of metal oxide photocatalysts [12], composites [13] and nanoparticles [14], etc. Using a low cost material [15] as agricultural wastes for toxic metals removal from wastewater has been investigated. Agricultural wastes consist of cellulose, hemi-cellulose, lignin, carbohydrates and some proteins which to be more effective adsorbent for heavy metal ions. Agricultural wastes have been found to be beneficial for metal sorption such as coconut [16], papaya seeds [17], neem leaves [18] banana peel [19], rice husk [20] and groundnut shell [4] have also been investigated in aqueous solutions [21] Preparation of agricultural wastes by applying nanotechnology give high qualities materials in, surface area, porosity, adsorption capacity, low cost and environmentally friendly [22].

The net dry matter in tea leaves (*Camellia sinensis*, *Theaceae*) contains mainly aromatic, carboxylate, hydroxyl, phenolic, and oxyl groups. This composition is responsible for its ion-exchange behavior [23]. Pomegranate peels (*Punica granatum L.*) containing considerable shares of polyphenols, lignin, and cellulose that consider as natural adsorbents to adsorb a number of heavy metal ions [24].

The recovery of the adsorbed Chromium on adsorbents has been carried out by burning the chromium-laden adsorbents at 500-600 ºC in presence of air. The adsorbents converted into carbon oxides, leaving chromium with silica. The silica was filtered out by dissolving the residue in distilled water, whereby Chromium goes into the filtrate. The percent recovery of chromium increases as the chromium adsorbed per unit weight of adsorbent increases and can reach 99% recovery [25].

The aim of this study was finding the optimum parameter as the particle size, dose, pH, contact time to remove the chromium from the polluted industrial wastewater to safe reuse in agricultural purposes by using the agricultural wastes as GTn and PPn. The ability of agricultural materials to adsorb metal ions has significant attention for the development of clean, cheap, and efficient technology for wastewater treatment.

2. Experimental

2.1 Materials

The chemicals used in this study were analytical grade as HCl, NaOH, and standard Chromium solutions**.**

2.2 Preparation of the adsorbents

Green tea (GT) and pomegranate peels (PP) wastes were collected from home consumption and then washed with hot distilled water at 85 °C to eliminate adhered particles from the surface and water-soluble material up to color removal. It is dried in a hot oven for 12 h at 105 °C to remove moisture then ground by a kitchen mill. The fine powder was passed throughout a sieve 0.25 mm; the passing powder was taken to re-grind it to obtain a nanopowder. Morphology and size of the Green tea (GT) and pomegranate peels (PP) wastes were investigated by high resolution transmittance electron microscope HRTEM, JEOL 3010.

2.3. Methods

The industrial effluent sample was obtained from El Roubiky City for Leather Industry, North of Badr City, and Cairo Governorate. The wastewater sample had a pH of 8.3 was measured using pH meter model WTW Series pH 720. Chromium ions concentration (142.31 mg/l) was measured in wastewater before using the adsorbents by spectrophotometer (model JENWAY6705UV/Vis) according to Willems et al. [26].

The experiments were performed in a series of flasks containing 100 mL of wastewater with an initial Chromium concentration of 142.31 mg/l and mass of adsorbents GTn or PPn. The mixtures were shaken for 1 h using a shaker. Then filterated using Whatman No. 1 filter paper to determine the concentrations of Chromium.

The effect of several parameters was studied, as pH value at 2, 6, 7, 8, 9, and 10. The pH was adjusted by adding a few drops of 0.1M NaOH or 0.1 M HCl solutions. Also, the effect of adsorbent doses 0.15, 0.3, 0.6, 1.0, 1.5, and 2g on the adsorption process was studied. The initial concentration used was 142.31mg/l in tanning wastewater. In addition, the effect of the contact time was varied between 30, 60, 90, 120, 150, 180, and 210 min at room temperature

The removal efficiency percentage of chromium ions can be calculated by:

$$Cr(VI)removal\% = \frac{C_i - C_f}{C_i} x100$$

Where C_i and C_f are the concentrations of heavy metal Cr (mg/l) before and after the treatment

Statistical analysis

Regression (Curve estimation) analysis was used to determine the relation between removal Cr % and all parameters under study using Statistical Package for Social Sciences program (SPSS) version 20 (IBM SPSS 2020).

3. Results and Discussion

3.1 TEM spectroscopy

TEM image of the prepared green tea spoil (GTn) and pomegranate peels (PPn) powder as shown in Figure 1. It is seen that the powder in nanosize with diameter ranged from 12 to 26 nm and 13 to 43 nm for GTn and PPn respectively. This reflects that the powder has a relatively large surface area for high capacity adsorption material.

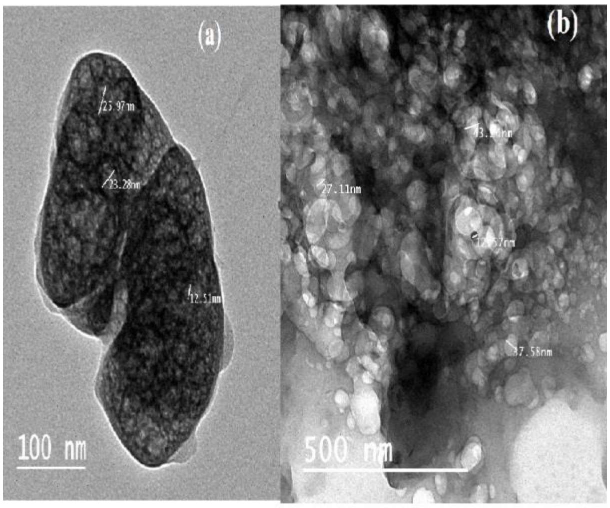

Fig. 1 TEM images of (a) green tea (GTn) and (b) pomegranate peel (PPn) nanopowders.

3.2. Effect of pH

Previous studies on heavy metals adsorption have shown that pH is the important factor affecting the adsorption process. The variance of pH is controlling adsorption of chromium ions from wastewater. Figure 2 shows that the effect of pH range from 2-10 on chromium ions removal efficiencies by both adsorbents green tea spoil (GTn) and pomegranate peels (PPn) nanopowders, by using 0.6 gm of adsorbent dose and 30 min contact time

The efficiency of adsorption rapidly decreased with increasing the pH value, owing to the removal capacity is highly related to the protonation of adsorbent materials [27].

The maximum percentage removal was found at pH 2 was 98.6 and 96.7% but the minimum percentage removal at pH 10 with values 65.25 and 62.20% for GTn and PPn, respectively, this may be due to repulsion between surface functional groups (OH^-) and chromium ions [28]. The chromium ions removal by GTn and PPn were decreased when pH was increased. The dominant species of Cr ions in solution are $HCrO^{-4}$, $Cr_2O_7^{2-}$, $Cr_4O_{13}^{2-}$and $Cr_3O_{10}^{2-}$ [29, 30] which could be removed primarily by electrostatically nature at optimum pH [31]. All sites of sorbent surface at GTn and PPn would be occupied by hydrogen protons which enhance the Cr ions interaction with binding sites by greater attractive forces at very low pH values [32, 33]. The total surface charges on GTn and PPn became negative and removal of chromium ions are decreasing when pH increasing [34]. Furthermore, in the case of high chromium ions concentration, the $Cr2O_7^{2-}$ ions precipitate at higher pH values [35]. GTn and PPn surface would be positively charged up to pH< 4, and the charge is varied in pH range 4–6 after that, the charge should be negative [36, 37].

Generally, the adsorption of chromium ions in aqueous solutions was highly dependent on the pH number. The use of GTn and PPn at the nanoscale increases the efficiency of removing chromium ions at all pH values

As shown in Figure 3 the relation between removal Cr % and pH was significant and highly correlated with 0.955 R^2 for both GTn and PPn.

3.3. Effect of contact time

The initial concentration affects the bonding with the adsorbents and gives the needed force to outdo the resistance of mass transfer from the liquid to a solid phase, so at a high initial concentration of Cr ions the adsorption rate increases [36]

Figure 4 shows the effect of contact time on the chromium adsorption using GTn and PPn from tannery wastewater. Although, was clear that increasing contact time increases the removal efficiency but at a specific time the percentage removal becomes almost constant [38].

It is found that the percentage removal of chromium ions at a contact time of 30 min was 98.6 and 96.7% [39] and was increased to about 99.8 and 98.1% with time at 210 min for GTn and PPn respectively. The rate of chromium removal was rapid in the first 30 min and then slowed down. The slow rate of chromium removal is may be occurred due to the electrostatic retardation or repulsion between the negative charges of the chromium ions in the solution and those adsorbed on the surface of GTn and PPn [28].

At high concentrations, the competitive dispersion of Cr ions increased at the adsorption sites available and its pores are closed so the Cr ions are prevented from passing deep into the absorbent pores. It is obvious from Figure 5 the regression between removal Cr % and contact time was significant and highly correlated

3.4. Effect of adsorbent dose

The different GTn and PPn doses (0.15, 0.3, 0.6, 1.0, 1.5, and 2 g/100 ml) were studied to see the dose effect on the removal of Cr ions as shown in Figure 6, keeping other parameters constant as pH and contact time.

The results showed that with an increase in GTn and PPn dose, the percentage removal of Cr was increased, then after that, it becomes nearly constant with 1g/100 ml adsorbent. Anyway, the unit mass removed of chromium ions was decreased with increasing in GTn and PPn dose [38].

After getting the maximum percentage removal of Cr 98.7 and 96.7% for GTn and PPn, respectively, using 0.6g adsorbent dose, percentage removal of Cr became more or less constant. The percent removal of Cr (VI) increases rapidly with increasing the adsorbent dose due to the greater availability of the exchangeable sites and increase of the surface area [32]. Similar behavior has been observed in previous studies and arises from the effect of interactions between ions and adsorbents [40]. This can be referred to the fact that the increase of the adsorbent dose leads to more vacant binding sites for the Cr (VI) ions and there will be an increase in the rate of removal. But there will be a very slow increase in the percentage removal after an optimum dose at 0.6 g/100ml, pH 2, contact time 30 min and wastewater concentration 142.31Cr mg/l which may be due to the equilibrium state between the adsorbent and adsorbate [41]. At a high adsorbent dose, the screening effect of an external layer of a cell blocks the binding sites from metal ions and thus results in lower metal removal per unit adsorbent [42]. Moreover, the maximum chromium removal efficiency was observed at 2 g/100 ml GTn and PPn dose of tanning wastewater at 142.31mg/l Cr concentration with values of 99.8 and 97.7%, respectively.

Figure 7 revealed that the regression between removal Cr(VI) % and the adsorbent dose was significant and highly correlated.

3.5. Applicability to tanning wastewater

The efficiency removal of Cr (VI) from aqueous solutions depends on particle size. The particles size used were at the nanoscale (12- 40 nm) as shown in Figure 1 at an initial adsorbate concentration of 142.31 mg /l. The maximum Cr removal efficiency was 98.6 and 95.5% for GTn and PPn, respectively at 30 min contact time, pH 2, and 0.6 adsorbent dose. On the other hand, after 210 min contact time, the Cr removals reach 99.8 and 96.7% for GTn and PPn, respectively.

Furthermore, the adsorption capacity of GTn and PPn depends on particle size, pore size, and chemical composition which control its interaction with polar and non-polar adsorbates active sites which establish the type of chemical reactions with other molecules. Using 1mg adsorbents gives a change to the possibility of nanoparticle size more than 0.6 adsorbent leading to increase removal efficiency to 99.98 and 98.95 % respectively. This is due to fine porosity and a large surface area of small particle size. So the adsorption increases, due to the transfer of the Cr ions through a short path inside the pores of the adsorbent particle [43].

An experiment was conducted to remove the chromium ions from tanning water at pH 7 using green tea and pomegranate peels nanopowder at adsorbent dose 1g/100 ml, contact time 210 min and initial wastewater concentration at 142.31mg/l, that the resulted of removal were 99.98 and 98.95 % of Cr (VI), respectively, as shown in Figure 8. From the above, it became clear that green tea spoil and pomegranate peels nanopowder are efficient in removing chromium ions from tanning wastewater. This may be due to the content of the polyphenolic compounds and the use of nanoscale residues led to an increased surface area and function groups that adsorb chromium ions from wastewater. The total polyphenol content of green tea spoil and pomegranate peels were 6.50 and 5.30%, respectively, this agreed with [24, 44- 46]. El- Nemr *et al* [36] are reported that the maximum percent adsorption of Cr ions were 86 and 82.5 % for tea wastes and pomegranate peels powders at bulk sizes and optimum conditions.

As evident from Figure 8 there was a significant and highly correlation between removal Cr (VI) % and contact time, using initial concentration 142.31 mg/l, pH 7, and adsorbent dose 1 g/100 ml onto (a) GTn and (b) PPn at different contact time. Furthermore, the particle size at the nanoscale plays an important role in the adsorption of Cr (VI) on GTn and PPn due to high surface area and high chemical activity.

Use of carbon from both green tea spoil and pomegranate peels nanopowder in the removal of Cr (VI) with concentration 142.31 mg/l from tanning wastewater led to removing more than 98%, as well as adsorb the hateful odor, eliminating the fungi which formed on the water surface, remove yellow color and remove the turbidity, so that the color of the wastewater turns into a clear transparent. Therefore, more research and study should be done in this case.

The use of agricultural waste for the treatment of wastewater containing toxic chromium ions in developing countries is an area that deserves interest.

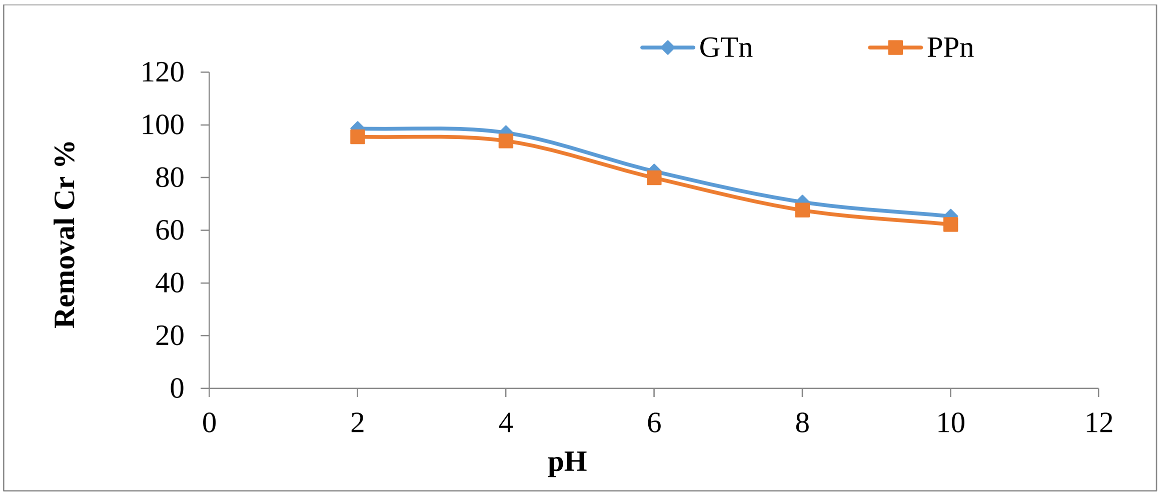

Fig. 2 Effect of pH on removal of initial Cr concentration (142.31 mgl^{-1}) onto GTn and PPn at contact time30 min using 0.6g/100 ml.

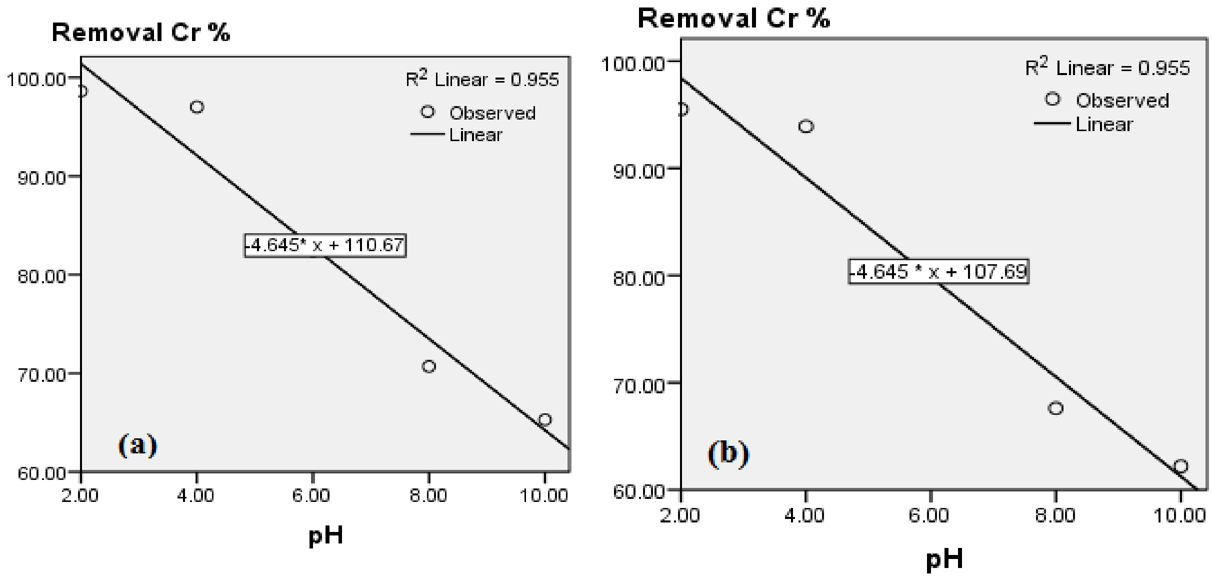

Fig. 3 The relation between the removal percent of Cr and pH at initial concentration 142.31mg/l, adsorbent dose 0.6g/100 ml and 30 min contact time using (a) GTn and (b) PPn

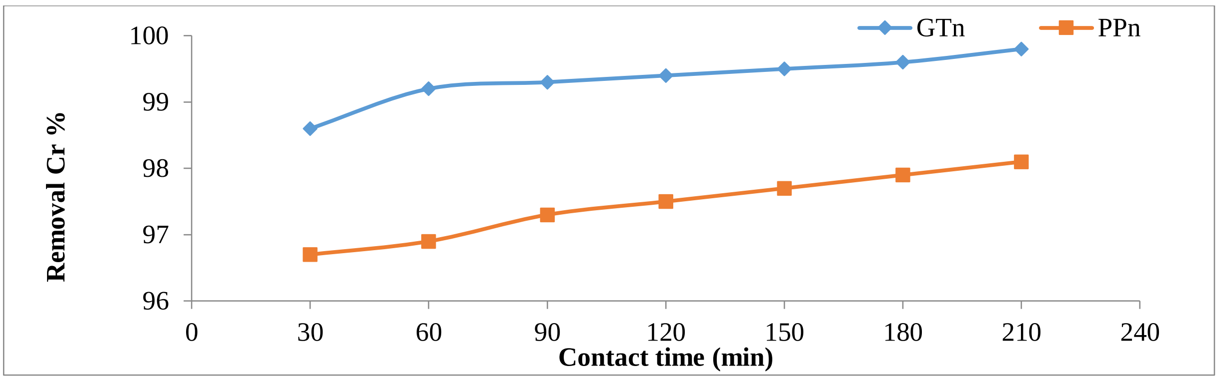

Fig. 4 Effect of contact time on removal efficiency with initial Cr concentration of 142. 31 mg/l using GTn and PPn at adsorbent dose 0.6 g/100 ml at pH 2.

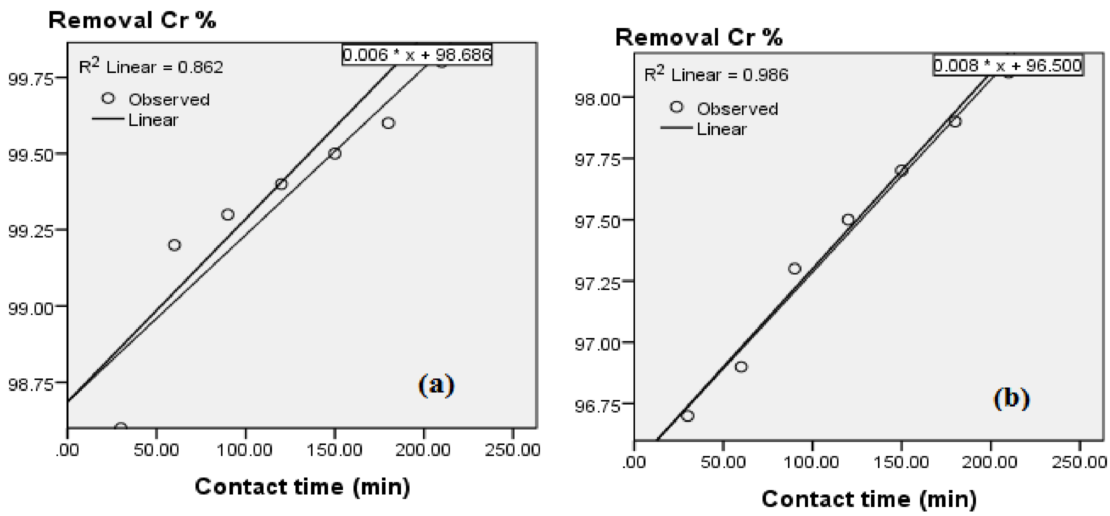

Fig. 5 The relation between removal Cr % and contact time at initial concentrations 142.31 mg Cr/l, 0.6 g/100 ml and pH 2 using (a) GTn and (b) PPn

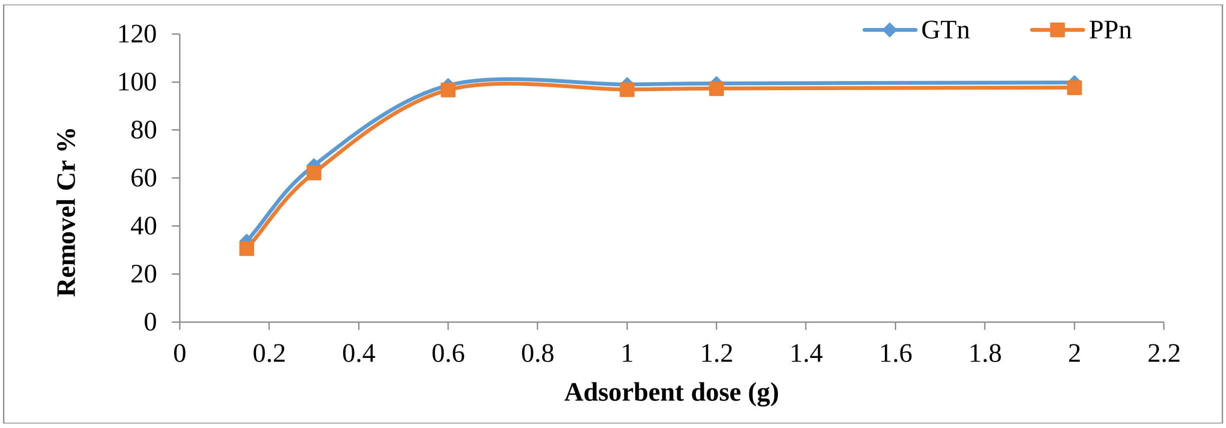

Fig.6 Effect of the adsorbent dose on the Cr ions removal, initial concentration 142.31 mg/l, contact time 30 min and pH 2.

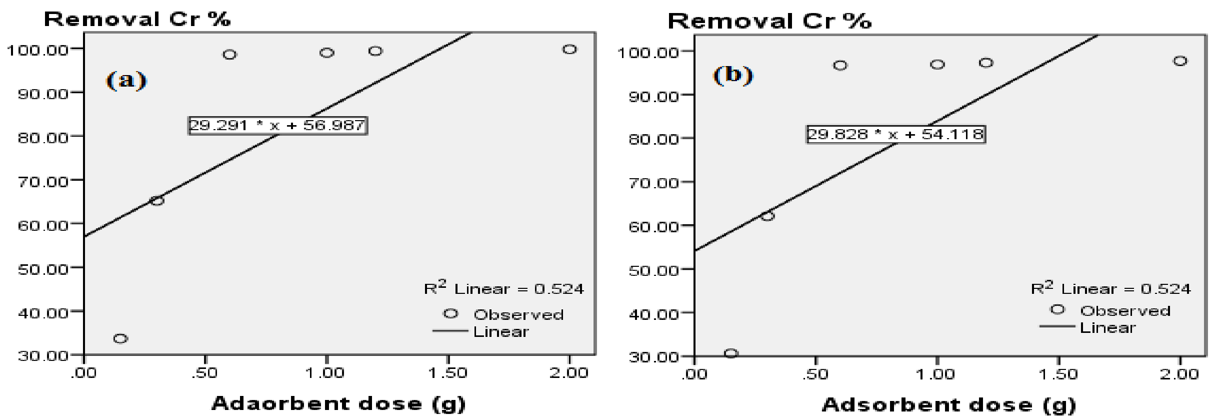

Fig. 7 The relation between removal Cr % and adsorbent dose at pH 2, contact, time 30 min and initial concentration 142.31 mg/l using (a) GTn and (b) PPn.

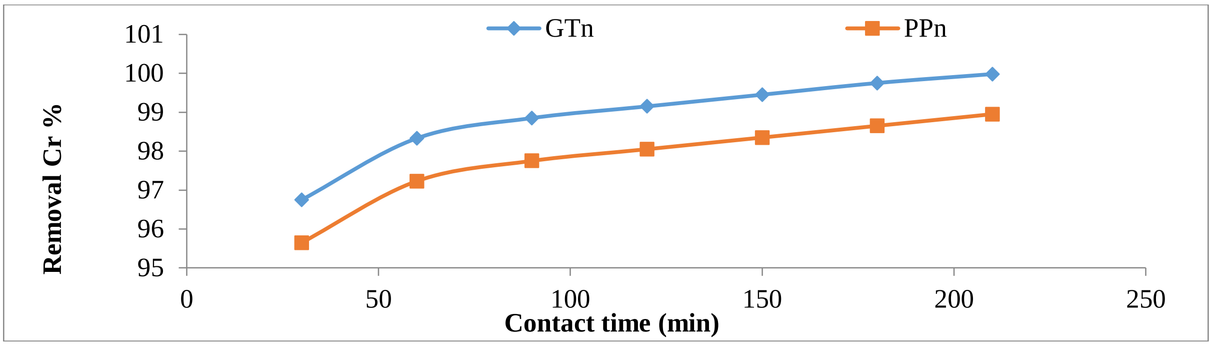

Fig. 8 Effect of different contact time on Cr (VI) ions adsorption from tannery wastewater at pH 7, contact time 210 min and adsorbent dose1 g/100 ml.

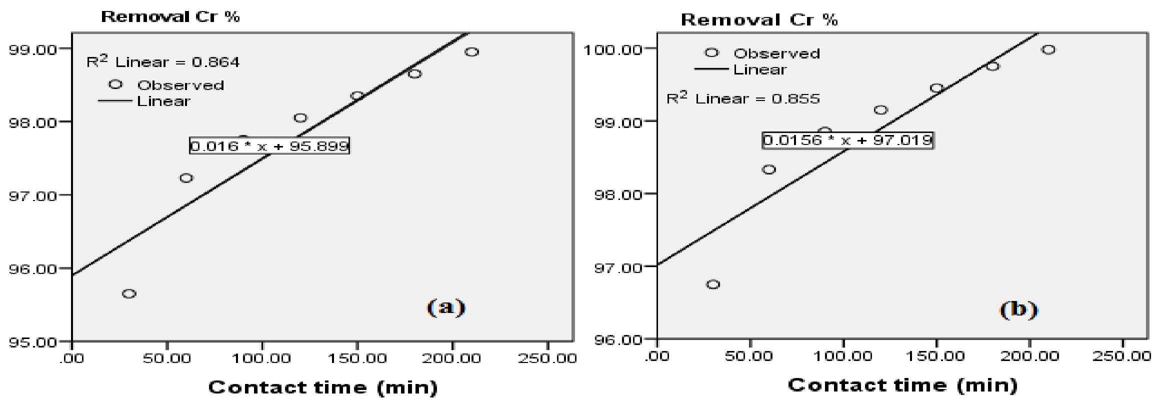

Fig.9 The relation between removal Cr % and contact time of tannery wastewater at pH 7 and adsorbent dose 1 g/100 ml using (a) GTn and (b) PPn.

CONCOLUSION

Could be concluded that green tea spoil (GTn) and pomegranate peels (PPn) nanoparticles are the effective and cheap adsorbents for the removal of Cr (VI) ions from tannery wastewater. Experiment results showed that at pH 7, 210 min contact time, and 1g/100ml adsorbent dose, the removal efficiency of Cr (VI) ions at initial concentration 142.31 mg/l in wastewater is 99.98 and 98.95% for both GTn and PPn, respectively.

3. Conflicts of interest

"There are no conflicts to declare

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الملخص العربي

استخدامات بعض المخلفات العضوية بحجم النانو لمعالجة مياه الصرف الصناعي

أدى النمو الصناعي إلى تلوث البيئه وتساهم المعادن الثقيلة في هذا التلوث التي تتراكم في المسطحات المائية وهذا يسبب مخاطر صحية جسيمة لجميع الكائنات الحية. ويعتبرالكروم أحد المعادن الثقيلة التي تستخدم في العديد من الصناعات ويوجد الكروم علي شكلين سامين الكروم (III) والكروم (VI). تم استخدام بعض المخلفات الثانوية الزراعية مثل تفل الشاي الأخضر (GTn) وقشور الرمان (PPn) بعد تحويلها الي جسيمات نانونية لتكون مفيدة لامتصاص المعادن. وجد أن امتصاص أيونات الكروم (VI) يعتمد على معايير مختلفة مثل جرعة الممتزات ودرجة الحموضة ووقت التلامس وحجم الجسيمات. هدفت هذه الدراسة إلى معالجة مياه الصرف الصناعية الملوثة بأيونات الكروم باستخدام المخلفات الزراعية وبتكلفة منخفضة لإعادة استخدامها بشكل آمن في الأغراض الزراعية.

أظهرت النتائج أن كفاءة إزالة أيونات الكروم (VI) كانت 99.98 و 98.95٪ لكل من تفل الشاي الاخضر GTn وقشور الرومان PPn على التوالي عند درجة الحموضة 7 و 210 دقيقة من وقت التلامس و 1 جم / 100 مل جرعة ممتصة. إن أستخدام هذه المخلفات الزراعية المتوفرة والقابله للتحلل من شأنه أن يحل بعض مشاكل التخلص بالإضافة إلى تقنية أستخدام مواد أرخص لامتصاص المعادن الثقيلة في مياه الصرف الصناعي