

St. Paul's Convent School
"Ironic" Saviour : green synthesis of iron nanoparticles for
degradation of dye and removal of metal ions

鐵除害神器

Introduction

Nowadays, many industries require the use of dyes and metal ions, such as the textile and electroplating industry. If the sewage is not properly treated, it will cause unbearable consequences to the environment and humans. In science research papers, we found that using iron nanoparticles is a safe and effective way to degrade some of these harmful dyes and remove some of the heavy metal ions. Since the traditional ways in synthesizing nano iron involve the use of hazardous chemical reducing and capping agents, such as sodium borohydride, disruptions to the environment may be caused. Therefore, our project aims to synthesize nano iron in a green way by making use of recyclable materials, such as Poinsettia Christmas flower leaves and hand warmer powder, to tackle water pollution problem.

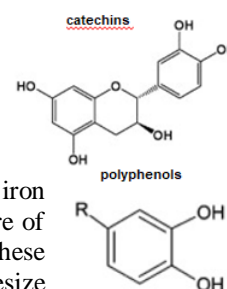
Objectives

In Part 1, we synthesized nano iron by reducing Fe^{2+} , Fe^{3+} and mixture of Fe^{2+}/Fe^{3+} using 6 types of leaf extracts and 3 types of tea, while we also synthesized iron nanoparticles by mixing hand warmer solutions with 3 types of Poinsettia (Christmas flower), 2 types of normal leaves and a type of tea. From these, we found out the best way for synthesizing iron nanoparticles of the greatest yield. In Part 2, we used the iron nanoparticles synthesized to degrade the following hazardous dyes - gentian violet, methylene blue, and malachite green. In part 3, we investigated the effectiveness of iron nanoparticles synthesized on the adsorption of Cu^{2+} , Ni^{2+} and $Cr_2O_7^{2-}$ by colorimetry and the removal of Pb^{2+} by gravimetric analysis. In part 4, we investigated the effectiveness of a filtration device in degrading the dyes and removing a mixture of metal ions.

Chemical Principles

Part 1 Synthesis of Iron Nanoparticles from Leaves, Tea and Hand Warmer Powder

The synthesis of iron nanoparticles is a reduction process of iron ions in iron(III) chloride or ammonium iron(II) sulphate solution into iron atoms with the naturally occurring reducing agents in leaves and tea, such as catechins, polyphenols and hydroxyl groups. On the other hand, hand warmer powder contains iron powder, which is oxidized to form a mixture of iron(II) oxide and iron(III) oxide during use. The mixture of iron(II) and iron(III) oxides will neutralize hydrochloric acid, forming iron(II) (III) salt solution. These solutions are added to leaf extracts to be reduced by the reducing agents in leaves as mentioned, to synthesize Fe nanoparticles.



Part 2 Degradation of hazardous dyes

Photo-catalysis of dyes takes place in the presence of UV light and iron nanoparticles. Under UV light, nano iron catalyzes the breakdown of water (H_2O) and oxygen (O_2) in air into hydrogen peroxide (H_2O_2). Hydroxyl radicals, which are highly reactive and unstable, will react with the dye molecules and oxidize them, resulting in dye degradation.

Part 3 Removal of metal ions

The nano iron is amphoteric in nature due to the presence of polyphenols, catechins and other functional groups on the surface of the iron nanoparticle. This greatly increases the surface area for the adsorption of metal ions. The development of surface charge on nano iron will give rise to an electrostatic attraction, which will cause the metal ions to be attracted to nano iron.

Part 4 Preparing a filtration device

Based on the results in parts 2 and 3, a filtration device was prepared using the catalysts and adsorbents with the best effects. The effectiveness of the device is tested by pouring the dyes - gentian violet, methylene blue, and malachite green, and a mixture of copper(II) nitrate, lead(II) nitrate and nickel(II) nitrate through the device.

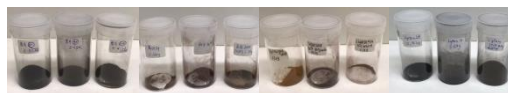
Experiment

Part 1 –Synthesis of Iron Nanoparticles from Leaves, Tea and Hand Warmer Powder

Leaves were ground using a mortar and pestle. The mixture was filtered using a sieve and deionized water was added in a 1:10 leaf-to-water ratio such that a 10% leaf extract solution was obtained. For tea, 500 cm³ of boiling water were added to 5 bags of tea, 10 g in total of tea leaves in the beaker. The procedure was repeated to obtain two beakers of 500 cm³ water and 5 tea bags each. The tea bags were left in beakers of boiling water for 30 minutes. The tea bags were removed and 300 cm³ of the tea infusion was extracted. After preparing the leaf and tea extracts, 0.2M $(NH_4)_2Fe(SO_4)_2(aq)$ or/and 0.1M $FeCl_3(aq)$ were added respectively. 1.0M $NaOH(aq)$ was added until pH becomes 5 and a persistent black precipitate is formed. The mixture was filtered under suction. The residues were dried in an oven overnight, which were then ground and weighed.

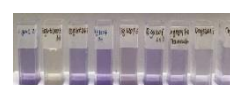


For synthesis of iron nanoparticles from hand warmer solution, 150.0 cm³ of 2M HCl was added to 10 g of each hand warmer powder. The mixture was heated for 2 hours and was left to stand overnight. The mixture was filtered and deionized water was added to obtain in a 250.0 cm³ volumetric flask to produce 250.0 cm³ treated hand warmer solution. Leaf extract of Poinsettia (Christmas flowers) was prepared using the same procedure as preparing for leaf extracts stated above. 12 cm³ of the extract was added to 80.0 cm³ treated hand warmer solution. 1.0M NaOH(aq) was added until pH becomes 5 and persistent black precipitate is formed. The mixture was filtered under suction and the residues were dried in an oven overnight, which were then ground and weighed.



Part 2 –Degradation of hazardous dyes

5 cm³ of 0.1% methylene blue, 5 cm³ of 1% gentian violet and 50 cm³ of 1% malachite green was diluted to 500.0 cm³ with deionized water respectively. 0.02 g of iron nanoparticles solid, which has been crushed into fine powder with pestle and mortar, or hand warmer powder was added to 20.0 cm³ of the 0.001% methylene blue, 0.01% gentian violet and 0.1% malachite green respectively. A control was also maintained without addition of iron nanoparticles. The mixtures were exposed to sunlight and were stirred for 2 hours using a magnetic stirrer. The mixtures were filtered, and the filtrates were centrifuged for 10 minutes, if necessary. The absorbance reading of the control and the treated solution were taken by a colorimeter using a 680nm filter for methylene blue, 580nm filter for gentian violet and 590nm filter for malachite green.



Part 3 –Removal of metal ions

For removal of Cu²⁺, Ni²⁺ and Cr₂O₇²⁻ ions, 20.0 cm³ of 0.01M CuSO₄(aq) or 0.01M NiSO₄(aq) or 0.01M K₂Cr₂O₇(aq) was added to 0.2 g of each adsorbent and the mixtures were stirred for 2 hours using a magnetic stirrer. The mixtures were then filtered. For Cu²⁺, 5.0 cm³ of each filtrate was mixed with 5.0 cm³ of 0.1M ethane-1,2-diamine. For Ni²⁺, 5.0 cm³ of each filtrate was mixed with 1.0 cm³ of 2.0M ammonia solution and 0.8 g of MSG powder. All the mixtures were poured into cuvettes respectively to obtain their absorbance in a colorimeter. For some mixtures with precipitate that cannot be filtered using a filter paper, the mixture was centrifuged for 10 minutes before taking the absorbance reading. For the removal of Pb²⁺, 30.0 cm³ of 0.01M Pb(NO₃)₂(aq) was added to 0.5 g of each adsorbent and the mixtures were stirred for 2 hours using a magnetic stirrer. The mixtures were filtered and rinsed with deionized water. 10 cm³ of 0.2M KI(aq) was added to each mixture. Mass of filter paper was weighed. The mixture was filtered and yellow precipitate was obtained as residue. After heating the filter paper in an oven, the filter paper was reweighed.



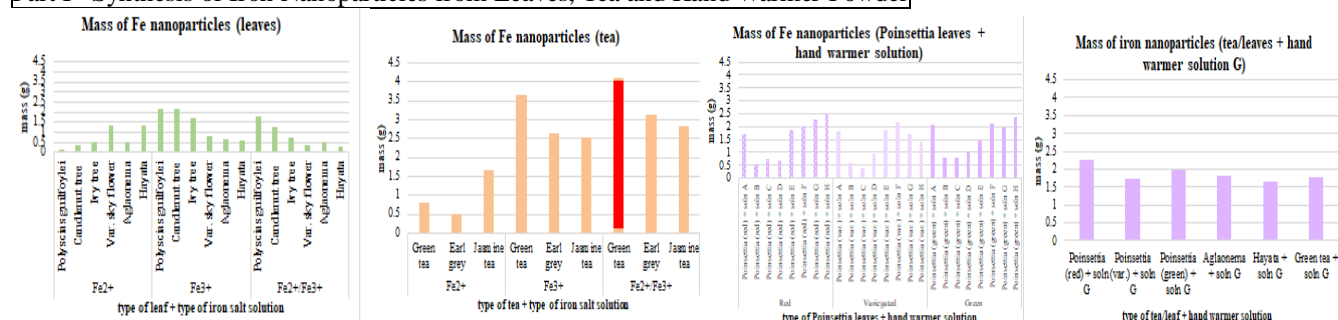
Part 4 Preparing a filtration device

Pieces of cotton wool were placed in an inverted plastic bottle. Selected used hand warmer powder and iron nanoparticles synthesized in part I were added on top of the cotton wool by layer. The plastic bottle was then held on a stand and a conical flask was put under the plastic bottle. Two tests were carried out on the filtration device to determine its effectiveness on degrading dyes and removing metal ions respectively. For effectiveness of degradation of dyes, 110 cm³ of 0.01% gentian violet was added to the conical flask through the filtration device. The absorbance reading of the control and the filtrate were taken by a colorimeter. The experiment was repeated using 0.001% methylene blue and 0.05% malachite green respectively. For effectiveness of removing metal ions, 20 cm³ of lead(II) nitrate, copper(II) nitrate and nickel(II) nitrate were mixed in a beaker. The mixture was added to the conical flask through the filtration device. 40 cm³ of sodium carbonate was added to the filtrate and the control respectively. Time was allowed for any precipitate formed in the mixture to settle. The mass of empty filter paper before filtering was measured. Then the mixtures were filtered under suction. The residues were dried in an oven, which were then weighed. The test was also repeated to test the sustainability of the filtration device.

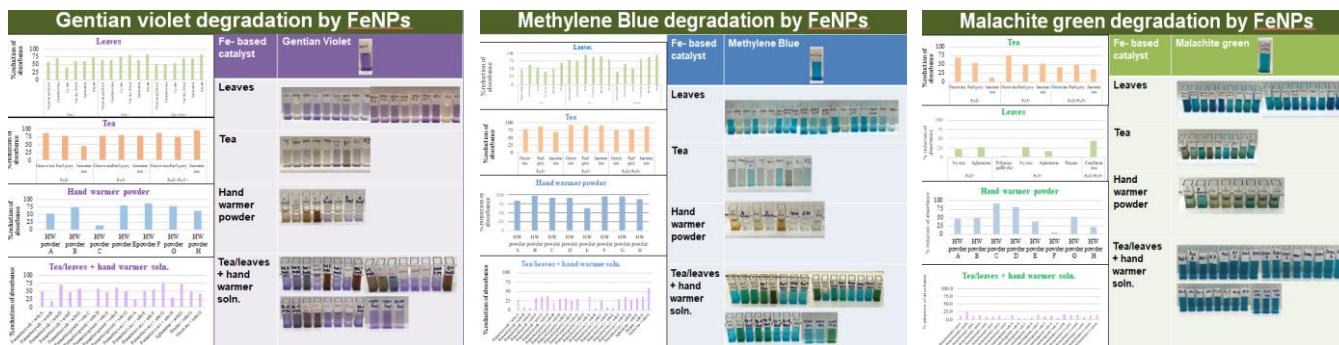


Results

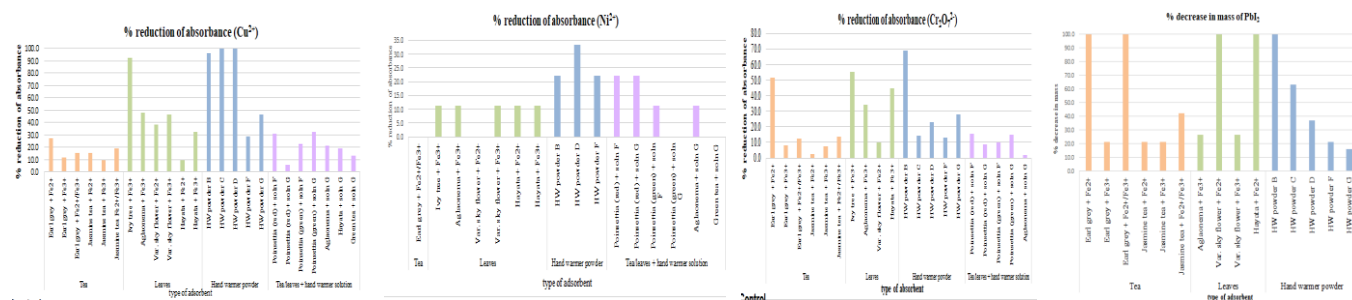
Part I –Synthesis of Iron Nanoparticles from Leaves, Tea and Hand Warmer Powder



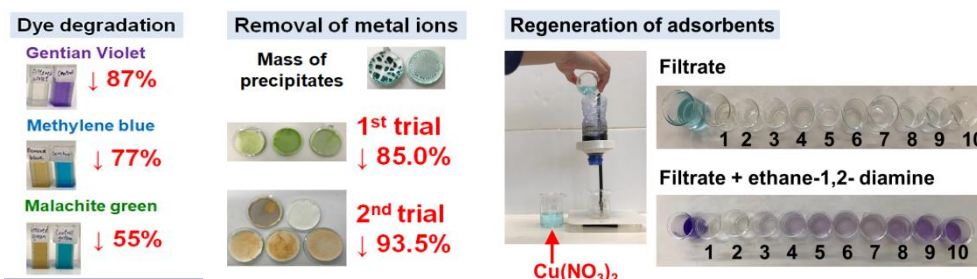
Part 2 –Degradation of hazardous dyes



Part 3 –Removal of metal ions



Part 4 Preparing a filtration device



Discussion

In part 1, comparing the yields of the 54 combinations of nano iron synthesized, we can conclude that Green Tea is the best in synthesizing the greatest amount of nano iron with the addition of a mixture of iron(II) and iron(III).

In part 2, nano iron synthesized when adding a mixture of iron(II) and iron(III) to jasmine tea is the best in degrading gentian violet; hand warmer powder B is the best in degrading methylene blue; while hand warmer powder C is the best in degrading malachite green. Overall, untreated hand warmer powder performs better in degrading dyes. The addition of iron(III) salt solution to tea and leaves shows better results.

In part 3, hand warmer powders C and D are the best adsorbents for removal of Cu^{2+} . For Ni^{2+} removal, the best adsorbent is hand warmer powder D. For the removal of $\text{Cr}_2\text{O}_7^{2-}$, the best adsorbent is hand warmer powder B. For the removal of Pb^{2+} , the best adsorbents are nano iron synthesised from adding Fe^{2+} to earl grey tea, variegated sky flower, hayata and hand warmer powder B. Overall, nano iron synthesized from hand warmer powders and leaves extract are the most effective in removing metal ions.

In part 4, the high percentage decrease in absorbance and mass of precipitate after carrying out colorimetry and gravimetric analysis on the filtrate shows the high effectiveness of our device.

Conclusion

To conclude, with emphasis on the effectiveness of a newly devised filtration device, the abilities of iron nanoparticles synthesized from greener and reliable materials in the control of water pollution by degrading dyes and removing heavy metal ions are shown.

