



Iron nanoparticles synthesized using green tea extracts for the fenton-like degradation of concentrated dye mixtures at elevated temperatures



Adam Truskewycz^{a,*}, Ravi Shukla^b, Andrew S. Ball^a

^a Centre for Environmental Sustainability and Remediation, School of Science, RMIT University, Bundoora, VIC 3083, Australia

^b Nanobiotechnology Research Laboratory and Centre for Advanced Materials & Industrial Chemistry, School of Science, RMIT University, Melbourne, VIC 3000, Australia

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ABSTRACT

Environmental harm caused by the release of textile dye contaminated wastewater from industry is an issue that requires immediate attention, particularly in developing countries. We herein demonstrate that green tea derived amorphous iron nanoparticles, in conjunction with Fenton like chemical processes, catabolise anthraquinone and azo dye mixtures that constitute a significant proportion of industrial dye waste. Iron nanoparticles, synthesized following a green tea mediated greener synthesis approach were able to degrade concentrated dye mixtures with over 90% decolourisation in only 20 min with 0.53 ppm nanoparticle concentration at a temperature of 70 °C. Detailed experimental degradation efficiencies were significantly pH and temperature dependant favouring lower pH values at temperatures between 70 and 90 °C, conditions which are commonly found in textile wastewaters. This research has shown the capacity for green tea synthesized nanoparticles to be used as a promoter for Fenton like dye degradation reactions. This rapid treatment approach may gain interest in the textile industries for dye waste remediation.

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1. Introduction

Environmental contamination with dye pollutants resulting from various textile staining activities is a concerning but an all too common practise. Pollution may stem from a variety of activities including but not limited to; out-dated waste management processes, accidental spills and cost cutting activities [1]. Furthermore, in developing countries, legal requirements and standards to address the release of pollution into the environment are less stringent and policed [2], leading to increased levels of pollution. In particular the impact of dyes in wastewaters is significant.

The applications for commercial dyes are vast and include the colorization of textiles, paper, leather, paper and plastics. Dyes are also used in food, drug, cosmetic and photochemical productions [3].

The two most common methods used to colour textiles are exhaust and continuous dyeing. Exhaust dyeing has two distinct phases; an absorption phase and a diffusion phase. Diluted soluble or partially soluble dyes are brought into contact with the fabric and are initially adsorbed onto the surface of the fibres. The heat of the dyeing vessel may then be increased to over 130 °C to facilitate the diffusion of dyes into the fibres. In continuous dyeing, concentrated dye solutions are applied to textiles and then subsequently fixed chemically or by thermal means in one simultaneous operation [4]. High temperatures therefore play a significant part of the dyeing process resulting in wastewaters that are also chemically but also thermally polluting.

Dye molecules are made up of colour bearing groups (e.g. an aromatic structure absorbing visible light) called a chromophore, and auxochromes which can aid in water solubility and strength for bonding to fibres [5]. Dyes can also be classified based on their mode of application to textiles such as: acid, basic, direct, dispersive, reactive, mordant, sulphur and vat dyes [6].

Azo dyes are the largest class of dyes with making up around 70% of all dyes produced (e.g. Direct Red 80 (DR80)). Anthraquinone dyes fall under the second largest produced dyestuff category (e.g. Remazol Brilliant Blue (RBB-R)) [7]. The difference between

Abbreviations: NP, nanoparticle; RBB-R, Remazol Brilliant Blue Dye; DR80, Direct Red 80 Dye.

* Corresponding author.

E-mail addresses: adam.truskewycz@rmit.edu.au, s3459597@student.rmit.edu.au (A. Truskewycz).

Azo and anthraquinone dyes lies within its structure. Azo dyes possess one or more azo chromophore (N=N) and bond between two or more aromatic rings whilst anthraquinone dyes possess the chromophore groups (=C=O & =C=C=) which form the anthraquinone complex [8].

Dye is a valuable commodity used to stain specific objects particular colours for aesthetic purposes. Nearly 40,000 dyes and pigments are listed making up over 100,000 different types of commercially available dyes. These dyes are produced in quantities of over 7×10^5 t per year with 10–15% of this entering the environment through industrial effluents [5].

One key attribute is that the dyes maintain their structure under harsh conditions and are therefore very resistant to biotic and abiotic degradation [9]. This makes it ideal for use within the textile industry. However, once these chemicals are released into the environment this same characteristic which makes it such a useful commodity becomes an environmental threat turning the dyes into persistent organic pollutants (POP's) [10].

Traditional treatments for water remediation have relied heavily on the transportation of water to remediation plants. These technologies have been deemed to be inefficient within reasonable timeframes and are often costly due to the energy requirements to pump water to the plant and the cost of infrastructure to facilitate remediation [11].

Monitored natural attenuation and bioremediation are other strategies employed to remediate contaminated sites. Although these technologies are cheap to implement, lengthy timeframes are required for remediation and many pollutants may be recalcitrant to breakdown from these biological entities [12].

Taha et al. have shown biosorption as a promising method for removing dyes from wastewater. In 12 h 81.14% of dye (100 ppm) could be removed from wastewater using inactivated *T. indica-seudaticae* at 55 °C [9].

The use of iron nanoparticles to catabolize pollutants and remediate contaminated sites is another technology which has been shown to be promising with research displaying degradation of a vast array of different dye classifications [13]. Although iron millimetric particles have been extensively studied and used, the shift from millimetric to nanoscale particles is the next frontier. With increased surface areas, NP's have the capacity to degrade pollutants orders of magnitude faster than their millimetric counterpart [14,10]. Preliminary work has shown that iron NP's remediate dyes to over 98% in 1 h at 30 °C. Furthermore, increases

in temperature have been shown to accelerate the rate of dye degradation using iron NP's [10].

The existing methods of iron nanoparticles synthesis are limited in their scope; as the reaction of an iron salt (e.g. iron (III) chloride) with sodium borohydride produces iron NP's but there are complications with this approach. Firstly, these particles quickly oxidise in the presence of air or oxygenated water reducing their stability and subsequent reactivity. Furthermore, these particles tend to agglomerate. In addition, sodium borohydride is an expensive and toxic compound which is therefore likely to contaminate the environment upon application. Therefore, extensive washing of the particles is required leaving behind sodium borohydride waste which must be disposed [15].

Commercially available nanoscaled zero valent iron particles (nZVI) from Toda Kogyo Corp are generated by reducing goethite and hematite using a thermal method in conjunction with hydrogen gas which not only requires large amounts of energy but also produces crystalline particles which have reduced reactivity compared to their amorphous counterpart. These particles are costly to manufacture due to high energy requirements and also have a tendency to agglomerate [13].

The use of green tea polyphenols to generate nanoparticles has been utilised for medical applications [16], but is also a promising approach for environmental pollution remediation because nanoparticles can be generated which possess the desired size, shape and crystallinity and the process can be considered an environmentally friendly, sustainable remediation approach. Polyphenols from the green tea reduce the iron salts to form iron nanoparticles and also cap the metal protecting it from oxidation (Table 1). Furthermore, green tea polyphenols are considered to be non-toxic and are therefore safer for the environment than sodium borohydride generated particles [17]. It is important to note that the use of iron salts for homogeneous Fenton processes has already been reported for pollutant remediation purposes. However, the main drawbacks of using salts include a small workable pH range (2.5–4.0), the formation of ferric hydroxide sludge at pH over 4 which can contain high concentrations of toxic pollutant degradation intermediates, and difficulty in catalysis recovery of iron [18]. Furthermore, since the hydroxyl radicals that are responsible for catalytic degradation process have a half-life of microseconds, a constant addition of iron salt solution is required to remediate pollutants which may be reintroduced into the system [19].

Table 1
Chemical composition of water soluble green tea constituents and their ability to interact with iron.

Component	Example of Chemical Constituents	Percentage Make-up (%)	Ferric Reduction Capacity?	Iron Binding Capacity?	Polyphenol?	Reference
Catechins	Gallocatechin, Catechin, Epicatechin, Epicatechin Gallate, Epigallocatechin; Epigallocatechin Gallate,	32–45.4%	Yes	Yes	Yes	[43,44]
Flavonoids (other than catechins)	Flavonols	7–14%	Yes	Yes	Yes	[45,46]
Methylxanthines	Caffeine	7–9%	Yes	Yes	No	[47]
Deposides	Theogallin	3–4%	Yes	Unknown	Yes	[48,49]
Organic Acids	Gallic acid, Ascorbic acid, Quinic acid	7–9.5%	Yes		No	[50,51]
Amino Acids	Aspartic Acid, Glutamic Acid, GABA, Alanine, Ammonia, Theanine, Threonine, Phenylalanine, Tryptophan, Lysine, Methionine, Isoleucine, Leucine, Valine	8–12.0%	Yes	Yes	No	[52,53]
Carbohydrates	Reducing sugars, lignin, Pectin	10–15%	Reducing sugars = Yes, Lignin and pectin = No	Yes	No	[54]
Minerals and Trace Elements	Ca, Mg, Cr, Mn, Fe, Cu, Zn, Mo, Se, Na, P, Co, Sr, Ni, K, F and Al	6–8%	No	Yes	No	[55]
Volatiles	1-Penten-3-ol, 1- α -Terpineol, Hexanal, 1,4-Dimethoxybenzene, Benzoic acid	0.02%	Unknown	No	No	[56,57]

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