An efficient heterogeneous Fenton catalyst based on modified iron rich soil for discoloration of Tartrazine simulated wastewater

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Abstract: An iron rich soil resource containing 67% Fe₂O₃ that is inactive towards the discoloration of tartrazine (TA) at neutral pH was used to prepare composites with charcoal obtained from aniline. The composites were obtained by heating this soil, aniline and sulphuric acid under air at 400°C and 600°C and the products (or catalyst) were denoted as Cal400 and Cal600, respectively. Cal400 and Cal600 were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Thermo-gravimetric Analysis (TGA), X-ray Diffraction, X-ray Fluorescence and Nitrogen adsorption Brunauer-Emmet-Teller (N₂-BET). Their catalytic activities were monitored by discoloration of tartrazine yellow dye and were performed by varying catalyst load (0.065, 0.125, 0.250, 0.375 and 0.500 g/L), H₂O₂ concentration (8, 16, 24 mol/L) and pH (5, 7, 8). The influence of NaCl, Na₂CO₃ and NaHCO₃ (1g/L) on the catalytic performance and the influence of initial pH on the iron leaching was also addressed. 94% of tartrazine was discolorized after 120 min of reaction with catalyst dose of 0.065 g/L, initial tartrazine concentration of 55 mg/L; H₂O₂ concentration of 8 mol/L and initial pH = 7. Leaching test indicated that the leached iron from the catalyst was less than 5 mg/L. Although all ions under study inhibited discoloration significantly, the inhibition efficiency of Cl⁻ is less than that of the others ions.

Keywords: Iron rich soil; Fenton-like process; Dye discoloration; Wastewater treatment.

1. Introduction

Dye pollutants from, printing and textile industries are important sources of environmental contamination. The effluents discharged from these industries are usually strongly colored, and their direct release into receiving water bodies could damage both aquatic life and human beings due to their toxic, carcinogenic and mutagenic effects [1,2]. Therefore there is an urgent need for the development of effective technology to remove dyes from wastewater and drinking water or water bodies.

Up to date, several methods such as adsorption, biological treatment and advanced oxidation have been studied for destruction of aqueous dyes [3-6]. Biological treatment would be economical, but requires long time to degrade dyes [7]. Adsorption is simple and effective, but it requires high operating costs associated with the generation of contaminant-spent adsorbents [8]. Compared to adsorption and biodegradation, advanced oxidation processes (AOPs) have been able to generate highly reactive radicals to disrupt a large number of hazardous pollutants without selection [9]. One of the most intensively studied AOPs is the process based on the Fenton reaction, which has its own unique advantages including high degradation efficiency, benign process, inexpensive materials and general applicability [10,11]. In the homogenous Fenton process, dissolved Fe²⁺ ions decompose H₂O₂ to OH⁻. Dissolved Fe³⁺ ions can also be utilized as the Fe²⁺ source. In this case, the process is called Fenton-like [12]. However, the industrial application of Fenton processes based on homogeneous Ferrous or ferric salts usually suffers several drawbacks such as formation of sludge as a result of the precipitation of iron hydroxide, narrow pH range, the production of iron-contained sludge and the catalyst deactivation by some iron complexing agents such as phosphate anions and some intermediate oxidation products.

To overcome these shortcomings of homogeneous Fenton oxidation, heterogeneous Fenton oxidation was found to be an efficient and cost-effective method. In this process, iron catalyst are immobilized onto solid supports (i.e., activated carbon, carbon nanotube, zeolite, clay) [13-16]. Also, insoluble iron oxides are used (goethite, magnetite and hematite), which can promote a heterogeneous Fenton process under less acidic conditions, and this has been largely explored by researchers in this area [17,18].

Iron oxide minerals are abundant in nature and are relatively inexpensive. They can be found in several parts of the world such as in Cameroon and Mexico [19, 20]. Moreover, they can be utilized in water treatment...
processes for removal of diverse contaminants by Fenton and photo-Fenton reaction [21]. However, iron oxide and other material based on iron oxide remain preferably in hydrophilic phase causing a low reaction rate [22]. For this reason, those materials need to be modified to generate hydrophobic regions on the catalyst surface. In this work, a natural iron rich soil (Nat) was treated with aniline to generate carbon on its surface in order to increase its catalytic properties. The effects of operating parameters such as the catalyst dosage, H₂O₂ concentration and initial solution pH on the discoloration of tartrazine were investigated. Given that dyes are usually used in association with various salts, the effect of some sodium salts were also examined.

2. Experimental

2.1. Materials

All chemicals used in this study were of analytical grade and were used without further purification. NaOH, H₂SO₄, and H₂O₂ 50%, NaCl, Na₂CO₃ and NaHCO₃ were obtained from Merck Co (Germany). The dye, C.I. Tartrazine (TA), was obtained from Sigma Aldrich (Germany), the molecular formula is C₁₄H₇₂Na₂O₁₄S₂, and molecular weight 534.4g/mol.

The natural iron rich soil denoted (Nat) was collected from Mbalam in the East region of Cameroon. The GPS coordinates of the sampling area are N: 02, 14,100-383105, E: 013, 56,926-247077 at an elevation of 812 m.

2.2. Preparation of catalyst

Approximately 5 g of soil was impregnated with 5 mL of 94% solution of aniline, after which 1 mL of 1:1(v/v) H₂SO₄ solution was added to accelerate charcoal formation. The solution was evaporated on a hot plate at 110°C and the solid obtained was ground and heated at two different temperatures in an electric furnace at 400 °C and 600 °C in air. The resulting products were labeled Cal400 (catalyst prepared at 400 °C) and Cal600 (catalyst prepared at 600 °C).

2.3. Characterization techniques

The chemical composition of Nat was determined by a Bruker-S4 Pioneer Wavelength Dispersive X-ray Fluorescence Spectrometer (WDXFS). The Fourier Transform Infrared (FT-IR) studies of Nat and catalysts in KBr pressed disks were performed using a Bruker Fourier Transform Spectrometer. The spectra were recorded in the wave number range from 4000 and 400 cm⁻¹. Thermogravimetric analysis TGA was done using an SDT2960 Mettler Toledo TGA/SDTA-851 Instrument. The recording was done under the following conditions: N₂ atmosphere with a heating rate of 10°C/min, gas flow of 30 mL/min and initial mass about 3.0 mg. X-ray diffraction patterns were recorded on a Siemens D5000 diffractometer with CuKα radioactive source. Finally, the pore volume, surface area and pore size distribution of the catalyst was measured through nitrogen purging at 77 K using a TriStar 3000 V6.05A equipment.

2.4. Catalytic performance Experiments

All experiments were carried out in a batch mode using conical flasks filled with 200 mL of the TA solution at a concentration of 55 mg/L. At a fixed pH, a given mass of the iron oxide catalyst (Cal400 or Cal600) and a given volume of 50% hydrogen peroxide (H₂O₂) were added to the TA solution. The mixture was maintained under constant agitation. During the oxidation reactions, 3 mL aliquots were withdrawn at selected time intervals, filtered and analyzed using a UV–Vis spectrophotometer (TECHNEL, S23A), at a tartrazine maximum wavelength of 420 nm. The withdrawn sample was returned into the conical flask to prevent any loss of contents. Equation (1) below was used to determine the discoloration efficiency (DE (%))

\[ DE(\%) = \frac{\text{C}_0 - \text{C}_t}{\text{C}_0} \times 100 \]

where C₀ is the initial dye concentration before discoloration and Cₜ is the concentration after reaction time t. The pseudo-first-order kinetic model (equation 2) was used to fit the discoloration of TA by Cal400 and Cal600. In order to compare the intrinsic catalytic activity of Cal400 and Cal600, the pseudo-first-order rate constants (k_app) were normalized by the total surface area per gram of catalyst as described by equation (3) [23].

\[ \ln \left( \frac{\text{C}_t}{\text{C}_0} \right) = k_{app} t \]  
\[ K_t = k_{m} \times \text{SSA} \]

where kₘ = k_app/catalysts concentration and SSA is surface area per unit mass of the catalyst. The presence of dissolved Fe was determined by colorimetric test.

3. Results and discussion

3.1. Mineralogical and physicochemical characterization of the iron rich soil

The chemical composition of Nat is given in Table 1 and it showed that the soil is extremely rich in iron, with a total Fe₂O₃ content of 67%. The soil also contains 20% of Al₂O₃ and the other oxides present in small amounts.

<table>
<thead>
<tr>
<th>Oxides</th>
<th>Fe₂O₃</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>P₂O₅</th>
<th>MgO</th>
<th>CaO</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>TiO₂</th>
<th>Cr₂O₃</th>
<th>I.I.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>67.16</td>
<td>20.5</td>
<td>1.58</td>
<td>1.09</td>
<td>0.05</td>
<td>0.05</td>
<td>0.15</td>
<td>0.08</td>
<td>0.33</td>
<td>0.016</td>
<td>8.75</td>
</tr>
<tr>
<td>LOI (%)</td>
<td>99.96</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 1

Chemical Composition of the iron rich soil sample

LOI = lost on ignition
The FTIR spectra of NAT, Cal400 and Cal600 are shown in Fig.1. The absorption bands at 3495 and 1634 cm⁻¹ in all the three spectra were assigned to the stretching vibration of H₂O molecule and bending vibration of O-H groups attached to the adsorbed water interlayer respectively [24]. In NAT, the absorption bands at 3095 cm⁻¹ corresponds to the stretching vibrations of structural OH groups of goethite and the intensive bands at 894 and 799 cm⁻¹ are assigned to Fe-O-H bending vibrations of goethite [20].

In FTIR spectra of Cal400 and Cal600 materials, there is displacement of the intensive bands from 894 and 799cm⁻¹ to 1100 and 1000 cm⁻¹ respectively which confirms the modification of Nat. Also the signals related to vibration of structural OH groups in Nat are no longer present in Cal400 and Cal600 due to dehydration and reduction of goethite. The band at 1444 cm⁻¹ in spectrum of Cal400 is assigned to bending vibration of C-O bonds indicating the presence of unburned carbon.

The TGA profile of the samples is shown in Fig.2. A total mass loss of about 9.3%, 47.3%, and 19.7% was observed for Nat, Cal400 and Cal600 respectively. Nat and Cal600 showed two well defined regions of mass loss while Cal400 showed three well defined regions. For all the samples the first region, between 50-100°C corresponds to a mass loss of approximately 1.8 %, 14.5% and 10.22% respectively, which may be due to the physically adsorbed water on the surface of the materials. For Cal400 the mass loss of 15.5% observed near 400°C is due to the decomposition of remaining aniline on the surface of the iron rich soil which was not completely decomposed when heated at 400°C. The mass loss of 18% and 9.5% respectively for Cal400 and Cal600 observed at 600 °C is due to the oxidation of carbon formed from aniline [25].

In order to identify the crystalline phases, the materials were characterized by X-ray diffraction. The XRD patterns for all samples are shown in Fig.3. XRD patterns of the natural soil (Nat) showed the following peaks at d-spacing of: 3.70A°; 2.71A°; 2.52A°; 2.21A°; 1.84A°; 1.70 Å which according to the standard powder diffraction data (JCPDS) are associated with hematite as a major crystalline structure. Also, the peaks at d-spacing of 4.20, and 2.51 Å are related to the goethite phase [26]. In the XRD pattern of Cal400 and Cal600 there is disappearance of the peaks associated to goethite and the appearance of a new peak in Cal600 at d-spacing of 3.21Å. This can be attributed to the diffraction of the (002) plane of the graphite structure because carbonization under high temperature resulted to carbon structures with some degree of graphitic order [27]. This showed that the deposition of carbon on the material surface also modifies the structure of the iron oxide.

The N₂ adsorption-desorption isotherms of Nat, Cal400 and Cal600 are shown in Fig.4. The shape of the isotherms seemed to be nearly type IV isotherm according to the IUPAC classification, indicating the presence of mesopores in the size range of 2–50 nm. BET surface area, total pore volume and average pore size of the Nat, Cal400 and Cal600 are summarized in Table 2. The BET surface area is of the order cal400 (63.26m²/g) > Nat (42.26m²/g) > Cal600 (6.03m²/g) indicating that the presence of charcoal on the surface of the soil increased the surface area when heated at 400°C, but decreased when heated at 600°C. This can be due to shrinkage and hardening of individual particles as well as the formation of agglomerates or clusters of particles through fusion (sintering). These processes may contribute to the reduction of the surface area. The pore size of cal400 decreased compared to that of Nat which is in favor of the catalytic property of cal400.
with observed cleavage constant results, after shown efficient attack diminished respectively.

3.2. Catalytic abatement of tartrazine

The experimental observations of tartrazine discoloration in the presence of Cal400 and Cal600 are shown in Fig.5. According to the results, the discoloration efficiencies after 120 min of reactions were 93.61 % and 48 % for cal400 and cal600 respectively. The discoloration of TA with Nat was not efficient at pH=7. This may be due to lower adsorption capacity of this soil at this pH (pH 7 > PZC (4.2) of Nat) or lower dissolution of iron at this pH. The pseudo-first-order rate constant for the discoloration of TA by Cal400 and Cal600 were 0.019 min⁻¹ and 0.002 min⁻¹ respectively. Normalizing these rate constants by the total surface area per gram of catalyst, we found values corresponding to 0.00053 and 0.00060 min⁻¹.L.m⁻² for Cal400 and Cal600 respectively. From this result, we observed that Cal600 had a higher discoloration rate constant normalized to surface area. Based on these results, Cal400 was selected for the catalytic tests.

The changes in the absorption spectra of TA solution before and during the discoloration process with Cal400 at different reaction times are shown in Fig.6. Before discoloration, the UV–Vis spectrum of TA is characterized by one main band in the visible region, with its maximum absorption at 420nm, which diminished very fast and disappeared almost completely after a reaction time of 120 min. This trend is due to the attack of the OH radical on the azo group of TA leading to the opening of the –N=O– double bond followed by cleavage of the long chain of conjugated π systems [28].

### Table 2

<table>
<thead>
<tr>
<th>Sample</th>
<th>$S_{BET}$ (m²/g)</th>
<th>Average pore size (nm)</th>
<th>Total pore volume (cm³/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nat</td>
<td>42.26</td>
<td>12.36</td>
<td>0.0891</td>
</tr>
<tr>
<td>Cal400</td>
<td>63.26</td>
<td>10.67</td>
<td>0.200</td>
</tr>
<tr>
<td>Cal600</td>
<td>6.03</td>
<td>26.50</td>
<td>0.064</td>
</tr>
</tbody>
</table>

3.3. Kinetic influence of operating conditions

3.3.1. Effect of Cal400 concentration

The effect of Cal400 dosage (0.065, 0.125, 0.250, 0.375, 0.5 g/L) at H₂O₂ concentration of 8mmol/L and initial pH of 7 on degradation of TA was evaluated (Fig.7). The results showed that the degradation process was very fast and the degradation rate increased with increase in catalyst dose until the dosage reached 0.375g/L. This reaction rate increased with the catalyst dosage from 0.065 g/L to 0.375 g/L because more H₂O₂ molecules might have been captured by increasing the catalyst dosage and be transformed to hydroxyl radical (•OH). Subsequently, the free radicals oxidatively damage and further mineralize the TA molecules. Generally, in heterogeneous Fenton system, when the catalyst is contacted with H₂O₂ solution, iron could be slowly dissolved into liquid phase to form a Fenton reactant, which is converted into iron ions. Kwan and Volker observed that organic matter decomposition rate is favored by increasing iron oxide concentration in heterogeneous Fenton like reaction [18].

**Fig.4.** Nitrogen physisorption isotherms of Nat, Cal400 and Cal600

**Fig.5.** Tartrazine discoloration in the presence of the Cal400 and Cal600. Conditions: [TA] = 55 mg/L, H₂O₂ = 24mM, [Cal400] = 0.500 g/L, [H₂O₂] = 8mM, and pH = 7

**Fig.6.** UV–vis spectra changes of TA in heterogeneous Fenton process. Conditions: [TA] = 55 mg/L, H₂O₂ = 24mM, [Cal400] = 0.065 g/L, and pH=7
However, reaction rate slightly decrease when cal400 concentration was increased to 0.5 g/L and could be ascribed to the inhibition effect caused by excess iron ions in the heterogeneous Fenton process that acted as scavenger. The kinetics of the discoloration of TA was simulated using a pseudo-first order kinetic model. The initial rate constants (k, min⁻¹) of the reaction were calculated to be 0.005, 0.012, 0.015, 0.024, and 0.019 min⁻¹ respectively for Cal400 dosage of 0.065, 0.125, 0.250, 0.375, 0.5 g/L. The slight drop in kinetic rate to 0.019 min⁻¹ corroborates the decrease in degradation rate for Cal400 dose of 0.5 g/L.

3.3.2. Effect of $H_2O_2$ concentration

$H_2O_2$ plays an important role as a source of $^•OH$ in Fenton reaction. In order to investigate the effect of $H_2O_2$ concentration, the initial concentration of $H_2O_2$ was varied between 8 and 24 mM. The result is presented in Fig.8. An increase in the concentration of $H_2O_2$ from 8 mM to 24mM caused an increase in the percentage discoloration from 52 % to 94%. This can be explained by the increase in the formation of OH$^-$ radicals which enhanced the discoloration efficiency of TA [29].

3.3.3. Effect of pH

It is well established that pH is an important parameter influencing the performance of both homogeneous and heterogeneous Fenton like processes [29]. So, three solutions with initial pH values of 5, 7 and 8 were studied (Fig.9) and the result showed that, all discoloration at each initial pH looked quite similar, which greatly differed from previous studies. For example, Hsueh et al. [30] reported that the degradation rate of RB5 (Reactive Black5) decreased sharply as the initial solution pH increased from 5.0 to 9.0. The reaction rate constants for pH of 5.0, 7.0 and 8.0 within 120 min were calculated by using pseudo-first-order model and were 0.0300, 0.0234 and 0.0254 min⁻¹ respectively. This observation was very important since it was previously accepted that one major drawback of the homogeneous Fenton was the tight range of pH for its reaction (that is close to 3).

Two aspects can be used to explain the result. On the one hand as a heterogeneous catalyst, the main advantage of Cal400 over the classic homogeneous catalyst (Fe$^{3+}$) is that Cal400 prevents the formation of iron oxide sludge hence expanding the effective pH range. A similar result has been reported that a heterogeneous catalyst (natural pyrite) performed similar activity in a wide pH range (3-11) in the Fenton reaction [31]. On the other hand, this may be due to the acid alkaline buffering capacities of the catalyst caused by Fe-polycations [$Fe_n(OH)_m(H_2O)_k^{(3n-m)}$] which were intermediate derivatives between the primary hydrolyzed products and the insoluble catalyst [32]. Under alkaline condition, most bases would be consumed by the Fe-polycations and as a result, the degradation of TA could occur effectively by the Fenton process only in acidic conditions [33].

3.4. Effect of inorganic salts on the discoloration of tartrazine

In order to confirm the role of OH$^-$ radicals on the degradation of TA molecules by Fenton like process, the effect of different scavengers including 1 g/L of

![Fig.7. Effect of Cal400 dosage on the discoloration efficiency of TA by heterogeneous Fenton-like process. Conditions: [TA] = 55 mg/L, [H$_2$O$_2$]=8 Mm and pH = 7.](image-url)

![Fig.8. Effect of H$_2$O$_2$ concentration on the discoloration efficiency of TA by heterogeneous Fenton-like process. Conditions: [TA] = 55 mg/L, [Cal400] = 0.065 g/L and pH = 7.](image-url)

![Fig.9. Effect of pH on the discoloration efficiency of TA by heterogeneous Fenton-like process, conditions: [TA] = 55 mg/L, [H$_2$O$_2$] = 8 mM, [Cal400] = 0.065 g/L.](image-url)
NaCl, Na₂CO₃ or NaHCO₃ on discoloration of TA was investigated (Fig.10). The results showed that inorganic salts had a negative impact on the discoloration of TA by Fenton like oxidation. The discoloration efficiency within 120 min reaction decreased from 91.7% to 66.5%, 25.4% and 5% respectively for NaCl, Na₂CO₃ and NaHCO₃. The inhibitive effect of these salts on the discoloration of TA can be explained by the stronger adsorption ability of the scavenger ions on the surface of catalyst than the TA molecules and also by the scavenging effect of Cl⁻, HCO₃⁻ and CO₃²⁻ ions to •OH. The chemical reactions are shown as below [34].

\[
\begin{align*}
\text{Cl}^- + \text{OH}^- & \rightarrow \text{ClO}^- + \text{H}^+ \quad (4) \\
\text{ClO}^- + \text{H}^+ & \rightarrow \text{Cl}^- + \text{H}_2\text{O} \quad (5) \\
\text{Cl}^- + \text{H}_2\text{O}_2 & \rightarrow \text{H}_2\text{O}^- + \text{Cl}^- + \text{H}^+ \quad (6) \\
\text{CO}_3^{2-} + \text{OH}^- & \rightarrow \text{CO}_3^{2-} + \text{OH}^- \quad (7) \\
\text{HCO}_3^- + \text{OH}^- & \rightarrow \text{CO}_3^{2-} + \text{H}_2\text{O} \quad (8)
\end{align*}
\]

![Fig.10. Effect of inorganic salts on the discoloration of TA. Conditions: [TA] = 55 mg/L, [H₂O₂] = 24mM, [Cal400] = 0.065 g/L, [salt] = 1g/L and pH=7](image)

### 3.5. Effect of initial solution pH on the Fe leaching from Cal400

To fully understand the discoloration behaviors of TA, it is necessary to determine the Fe concentration in solution versus time. Therefore, the Fe leaching from Cal400 can be measured, and the stability of the catalysts can be estimated at various initial solution pH. Fig.11 shows the effect of initial solution pH on Fe leaching from the Cal400 during the discoloration process. As observed, the initial solution pH significantly affected the Fe leaching from the Cal400. Fe concentration decreased with increase in pH and for each initial pH, the Fe concentration in solution increased from low level to a peak value followed by a decrease. For example at pH 5, there was an increase in Fe concentration from 2.34 to 2.55 and then a drop to 1.93 mg/L. This can be explained by the fact that Fe leaching is associated with some reaction intermediates such as oxalic acid that could capture Fe ions to form Fe complexes, which goes into solution. When the concentration of the intermediates attain a maximum value, the Fe concentration also exhibits a peak value and when the intermediates are mineralized into CO₂ and H₂O, the Fe ions return to the surface of the Cal400 [35]. After 120 min the total amount of iron dissolved in solution were 1.93, 1.89, 1.72 mg/L for initial solution pH of 5.0, 7.0 and 8.0 respectively. All the values were well below the standard of Environmental Quality (Sewage and Industrial Effluents) of 5 mg/L [29].

In order to evaluate the contribution of the homogenous reaction during the overall heterogeneous Fenton reaction, an experiment was carried out using only the aqueous iron ions produced by Cal400 dissolution at pH 7.0 as the catalyst. This experiment showed that the homogenous Fenton reaction resulted in 66% color removal. This indicated that most TAs were degraded by •OH produced from dissolved Fe(III) and not by the surface catalyzed process [36]. This led to the conclusion that the role of soluble, leached iron from Cal400 is significant in discoloration.

![Fig.11. Fe concentration in solution during discoloration of TA at different pH. Operating conditions: [TA] = 55 mg/dm³, [H₂O₂]ᵦ = 8Mm, pH 7 and [Cal400] = 0.5g/dm³.](image)

### 4. Conclusion

Aniline was carbonized over the natural iron rich soil surface at 400°C and 600°C in air and was used as Fenton-like catalysts for the degradation of Tartrazine solutions at various operating conditions. The Cal400 catalyst exhibited a high catalytic activity and almost 94% discoloration of 55 mg/L tartrazine was attained at 120 min reaction time. Though the initial pH of solution was varied up to 8.0, the catalyst was still efficient both in neutral and alkaline solution. The activity obtained under the homogeneous conditions shown by the iron leached conditions was higher than the heterogeneous conditions. Therefore, the oxidation mechanism was mostly homogeneous. The presence of radical scavengers inhibited discoloration significantly. Overall, this extremely cheap, eco-friendly material, used as catalyst in Fenton-like oxidation of dye is promising and its performance was comparable or even better than data reported in literature using more complex and...
expensive catalysts. This study showed that modified iron rich soil from East Cameroun can be used as a cheap efficient Fenton like catalyst for the treatment of organic dyes containing wastewater.

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