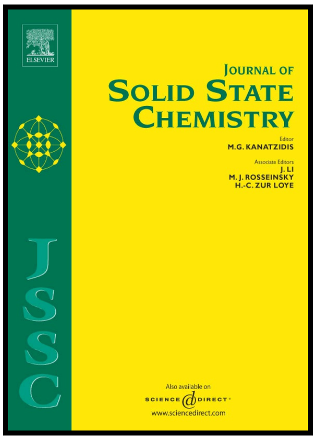

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Colour and toxic characteristics of metakaolinite–hematite pigment for integrally coloured concrete, prepared from iron oxide recovered from a water treatment plant of an abandoned coal mine

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Abstract

A metakaolinite-hematite (KH) red pigment was prepared using an ocherous iron oxide sludge recovered from a water treatment plant of an abandoned coal mine. The KH pigment was prepared by heating the kaolinite and the iron oxide sludge at kaolinite's dehydroxylation temperature. Both the raw sludge and the KH specimen were characterised for their colour properties and toxic characteristics. The KH specimen could serve as a pigment for integrally coloured concrete and offers a potential use for the large volumes of the iron oxide sludge collected from mine water treatment plants.

Keywords: mine water; Iron Oxide; metakaolinite; pigment; concrete

1. Introduction

Many of the coal mines in the South Wales Coalfield (UK) have been abandoned and perennially discharge large volumes of ocherous water that require treatment via settlement lagoons and reed beds, before being discharged into local river systems. The removal of the ocherous sludge generated from the mine water treatment systems is a major concern for the efficient operation of the treatment systems. The sludge also contains contaminants such as arsenic, cadmium, lead and zinc which can cause major environmental problems. The UK Coal Authority estimated that up to 380,000 wet tonnes (5% solids) of ferric hydroxide has accumulated within 60 (approximately) UK mine water treatment systems [1]. The sludge can also be converted as a sorbent in environmental applications such as phosphate and metal ion removal from water. The investigation into the potential resource application of ferric oxide would also reduce the volume of iron oxide sent to landfill and reduce disposal costs for the Coal Authority [1]. The iron oxides can be used to prepare yellow, red and brown pigments dependent upon the chemical state of iron. There have been successful attempts in the UK and the USA to reuse the iron oxide recovered from the mine water to produce inorganic pigments [1-4]. The present study introduces a new clay-based pigment prepared using kaolinite and iron oxide recovered from mine water and discusses the colour properties of the raw sludge. In the UK, the Coal Authority have undertaken a full-scale trial in partnership with a local brick manufacturer and utilised the entire output of sludge from the Dawdon Mine Water Treatment Plant, equating to 300 tonnes of dry ferric hydroxide per annum [1]. Goethite (α-FeOOH), is the predominant form of iron oxide and it is present in the mine water from abandoned coal mines. Though all iron oxides have high tinting and pigmenting strength, hematite shows far greater pigmenting characteristic than goethite [5].

Clay-iron and silica-iron based materials have been investigated as geopolymers and alternative construction materials such as ecological bricks, hematite-silica pigments and hematite-aluminosilicate gels [6-8]. Kaolinite, the 1:1 phyllosilicate whose thermal properties can be exploited to prepare composite materials [9-10]. Owing to its pozzolanic property, Metakaolinite (Calcined kaolinite) can be blended with cement to increase the strength and durability of cement as well as cement's resistance to sulphate and chloride [11-13]. The kaolinite is largely used as a pigment filler and extender in water-based interior latex paints. Calcined and delaminated kaolinites (heated above 600 ºC) are used extensively in interior water-based paints [14]. The calcined kaolinite can act as (i) a filler and extender for pigments produced from mine water sludge; (ii) a host for retaining the contaminants; and (iii) a good pozzolanic material. The present study is an attempt to produce a kaolinite-based pigment for integrally coloured concretes using iron oxide recovered from abandoned mine water treatment plants. The study shows the preparation, colour properties and toxic characteristics of metakaolinite-hematite (KH) specimen and the iron oxide sludge recovered from mine water.

2. Materials and methods

A commercially available kaolinite was procured and identified by X-ray diffraction analysis. The iron oxide sludge was collected from the Vivian ‘Six Bells’ mine water treatment system, Blaenau Gwent, UK (Referred as Raw FeOOH). Stoichiometric mix (Eq. 1) of ground and oven dried (100 ºC) kaolinite (<75µm) and goethite (<75µm) were heated at 600ºC for 12h in a furnace with temperature control of ± 5˚C. The resulting material, metakaolinite-hematite red pigment (hereafter referred as KH) was characterised for its colour and toxic properties. The reaction is based on the salt-induced kaolinite hydroxylation phenomena [13].

$$Al_2O_3.2SiO_2.2H_2O\ +2FeOOH+\ \overset{\blacktriangle}{\rightarrow}\ Al_2O_3.2SiO_2.Fe_2O_3+3\,H_2O\uparrow \qquad \text{(Eq. 1)}$$

(Kaolinite) (Goethite) 600ºC (Metakaolinite-hematite)

The elemental composition of kaolinite and Raw FeOOH specimens were measured using an Olympus X-5000 energy dispersion x-ray fluorescence spectrometer. The X-ray fluorescence spectrometer was calibrated for kaolinite using a china clay (CERAM Research catalogue no. AN 41; Supplied by Bureau of Analysed Samples Ltd., Newham Hall, Middlesbrough, UK) reference material and validated with the same material as well as a ball clay (CERAM Research catalogue no. 2CAS1) reference material diluted with analytical grade boric acid. The calibration for Raw FeOOH was carried out using a furnace dust (Euro-Standard 871-1 Furnace dust) and validated with the same material diluted with boric acid. The infrared absorption spectrum was obtained using a PerkinElmer Spectrum One spectrometer configured with attenuated total reflectance sampling unit with diamond crystal. Thirty scans were collected in the spectral range of 4000 cm^{-1} to 400 cm^{-1} with a resolution of 4 cm^{-1}. Background spectra were collected before each sample measurements. Powder X-ray diffraction (PXRD) patterns were recorded with a Philips X'pert Diffractometer (Ni-filtered Cu Kα radiation, λ = 1.5418 Å). PXRD data for Rietveld refinement of the structures were collected at room temperature employing the same diffractometer in the 2θ range of 10 – 100^o with a step size of 0.02^o and step duration 50s. The PXRD patterns were refined employing the General Structure Analysis System (GSAS) program [15]. A ninth order cosine Fourier polynomial for the background, zero, LP factor, scale, pseudo-Voigt profile function (U, V, W and X), lattice parameters, atomic parameters, and U_{iso} (total 23 parameters) were used in the refinement. The diffuse reflectance spectra for all the powdered samples were recorded on a PerkinElmer Lambda 35 UV – vis double beam spectrometer over the spectral region of 300– 1000 nm. The diffuse reflectance was converted to absorption using Kubelka-Munk equation.

$$F(R) = (1-R)^2/2R \qquad \text{(Eq. 2)}$$

, where F(R) is the Kubelka-Munk function and R is the reflectance.

Room-temperature solid-state emission was recorded for all the samples using a PerkinElmer Fluorescence spectrometer, (U.K. model no. LS55). CIE-1931 chromaticity coordinates were calculated using Gocie software [16]. SEM images of gold-coated samples of kaolinite (Acceleration Velocity (AV) = 10 kV, 21196 times magnification (Mag) at a probe working distance (WD) of 5.9 mm), Raw FeOOH (AV= 10 kV, Mag = 24,000 times and WD = 10.4 mm) and KH (AV= 10 kV, Mag = 24,000 times and WD = 10.5 mm) were recorded on a FEI ESEM Quanta 200 instrument with secondary and backscattered electron detector (Everhart-Thornley detector). Toxic Characteristic Leaching Procedure (TCLP) was adopted from Environmental Protection Agency method 1311[17]. The major cations and trace ions concentrations in the TCLP extracts were measured using a PerkinElmer Optima 2000 inductively coupled plasma optical emission spectroscopy (ICP-OES). The concentrations of water soluble anions were measured using a Dionex IC2000 ion chromatography configured with hydroxyl based anion retention column.

3. Results and discussions

3.1 Chemical properties of Kaolinite and Raw FeOOH.

The kaolinite was identified by X-ray diffraction (XRD) analysis (Fig 1b). The kaolinite composed of 45.4% SiO_2, 39.8% Al_2O_3, 2.4% K_2O, 0.9% FeO and 0.11% CaO. The PXRD pattern of kaolinite indicated the presence of illite phase as an impurity. Rietveld refinement was performed on the PXRD pattern of kaolinite by referring the crystal structure (Space group: *C1*, a = 5.154 Å, b = 8.942 Å, c = 7.401 Å, α = 91.69°, β = 104.61°, γ = 89.82°) reported by Neder et al [18]. As Rietveld refinement fit resulted in a fit with acceptable reliability factors, the illite impurity phase is assumed to be less than 5 mole percentage (Fig 1.a). The iron oxide sludge was identified as goethite by XRD (Fig 1c). X-ray fluorescence spectroscopy analysis revealed that the sludge contains 48% Fe, 3.68% Ca, 1.47% Mg, 0.5% Al, 0.11% Mn and 0.013% K.

3.2 Microstructural and colour properties of Raw FeOOH and KH specimens

The lattice band (Fe-O symmetric stretch) at wavenumber 599 cm^{-1}, which is normally observed at 630 cm^{-1} for well crystalline goethite indicating poor crystallinity or the possibility of aluminium isomorphous substitution in the Raw FeOOH [19-20] (Fig 2). The characteristic OH bending bands of goethite were identified at 890 cm^{-1} and 796 cm^{-1} [21]. The absence of kaolinite peaks in the XRD pattern obtained from the KH specimen shows that the kaolinite transformed as metakaolinite which is amorphous in nature and the peaks obtained are attributed to hematite (Fig 1d and 1e). The absorption bands identified at 524 cm^{-1} and 439 cm^{-1} are attributed to lath or platy shaped hematite particles [22] (Fig 2). However, the adsorption peak detected at 524 cm^{-1} is very close to the Al-O-Si deformation peaks of kaolinite [23] (Fig 2). The Scanning electron microscope (SEM) image of the KH specimen illustrated the collapsed kaolinite incorporated with hematite (Fig 3d & 3f).

Kaolinite showed a UV-spectrum with almost no absorption while Raw FeOOH and KH showed similar spectra with a broad absorption band ranging from 300 nm to 600 nm (Fig 4a). All the expected Fe^{3+} transitions are believed to be covered by this broad absorption band.

The clear assignment of these transitions following Sherman and Waite [24] is given in Table 1. There are two comparatively low absorption bands in the region from 600 nm to 900nm. The peaks around 650 nm in the spectra of Raw FeOOH and KH are assigned to $^6A_1 \rightarrow ^4T_2(^4G)$ transition. The transition $^6A_1 \rightarrow ^4T_1(^4G)$ occurs at 950 nm and 850nm in Raw FeOOH and KH respectively. This low absorption in the red region of the visible spectrum is responsible for the red colour of both the samples.

Variation of red hue (red ochre for Raw FeOOH and red for KH) can be understood by analysing the band gap of both samples. From the Tauc plots given as inset in the Figure 4a, it was noted that the goethite showed a band gap of 1.93 eV and the KH specimen showed a band gap of as 1.92 eV (indirect band gaps). The small difference in the band gap might have induced the difference in the colour [25]. The band gap energy of KH specimen is consistent with the reported indirect band gap of 1.90 eV in haematite [26]. Logically the Raw FeOOH (goethite) is yellow in colour (Fig 3a: iron oxide sludge dried), but along with the different band gap the aluminium isomorphous substitutions or particle size could be the reason for the redness shown in the colour coordinates (Fig 4b) [19-21]. The colour coordinates obtained for the KH and goethite samples fall in the red and reddish-yellow region of CIE chromaticity diagram, respectively (Fig 4b).

3.3 Toxic characteristics of KH specimen

The KH sample was characterised for water soluble contents, alkali resistant and toxic chemicals [17 and 27]. The TCLP results reveal that very marginal amount of contaminants were released (Table 2). The release of contaminants can be further reduced by the cementation property of the pigment (KH). The change in structural property could be the reason for the very low dissolution of Fe from KH samples at low pH condition (water pH was modified by diluted hydrochloric acid; The solid: water ratio was 1g:250 mL). Calcium was the only major cation leached out from the KH sample (Fig 5a and 5b). Thirteen mg of calcium per g of KH (which is equivalent of 3.25% limestone) was leached out at pH around 2.3. The maximum permissible limit of limestone content in Portland cement is 5% [28]. Considering the high alkaline pH of cement, the calcium leaching out from the pigment would possibly end up in the pozzolanic reaction to form the calcium-silicate-hydrate gel [29]. The concentration of sulphate was around 0.087% mass of KH specimen (Table 3) which is well within the prescribed limit of 5% mass of the original pigment specified in American Society for Testing and Materials (ASTM) C979/C979M − 10 [27]. The other major anions concentrations leached out in water also marginal (Table 3). There was no visual colour change in alkali treated KH specimen (Fig 3b) compared with KH specimen (Fig 3c).

4. Conclusions

The present study demonstrates the colour properties of the iron oxide recovered from a water treatment system of an abandoned coal mine (Raw FeOOH) and the pigment (KH). The KH pigment, the product of goethite-kaolinite reaction at 600˚C, can be a potential pigment for integrally coloured concrete. The metakaolinite presence in the KH pigment could improve the mechanical properties of the concrete. Apart from the colour properties, the KH pigment did not show any toxic characteristics. The work further demonstrates that the available iron oxide material from the water treatment plants of abandoned coal mines could be used for preparing iron-based composite materials whilst reducing the volume of iron oxide sludge sent to landfill from mine water treatment plants. The influence of this kind of pigment on the strength properties of concrete must be investigated in the future.

Acknowledgements

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Table 1. Assignment of transitions covered within 300 nm-600 nm broad peak

Transition	**Peak position in goethite (or) Raw FeOOH (nm)**	**Peak position in KH (nm)**
$^6A_1$+$^6A_1$→$^4T_2(^4G)$+ $^4T_2(^4G)$ Double excitation due to Fe-Fe pair	480	516
$^6A_1$→^4E,$^4A_1(^4G)$ & $^6A_1$→$^4T_2(^4D)$ (Could not be resolved)	~422	~419
$^6A_1$→$^4E_2(^4D)$	350	Not resolvable
$^6A_1$→$^4T_1(^4P)$	316	320

Table 2. TCLP results of KH and Raw FeOOH specimens

Element	mg/g of KH	mg/g of Raw FeOOH
As	ND	0.0017
Cd	ND	ND
Cr	ND	ND
Cu	0.00018	0.0011
Mo	0.00002	0.0001
Ni	ND	ND
Pb	0.0002	0.0003
Sb	ND	0.0003
Se	ND	0.0009
Zn	0.0022	0.003
Al	0.00196	ND
Li	0.00008	ND
Ti	ND	ND
V	ND	ND
B	0.0002	ND
Sr	0.012	0.085
Tl	0.0006	0.00012
Co	ND	ND
Ag	ND	ND
Be	ND	ND
Ba	0.0006	0.003
Bi	ND	ND

ND-Not determined or less than the detection limit

Table 3. Water soluble major anions at KH specimen's natural pH of 8.3

Element	mg/g of KH Specimen
F^-	0.85
Cl^-	2.32
SO_4^{2-}	0.87
NO_3^-	0.31

Figure 1

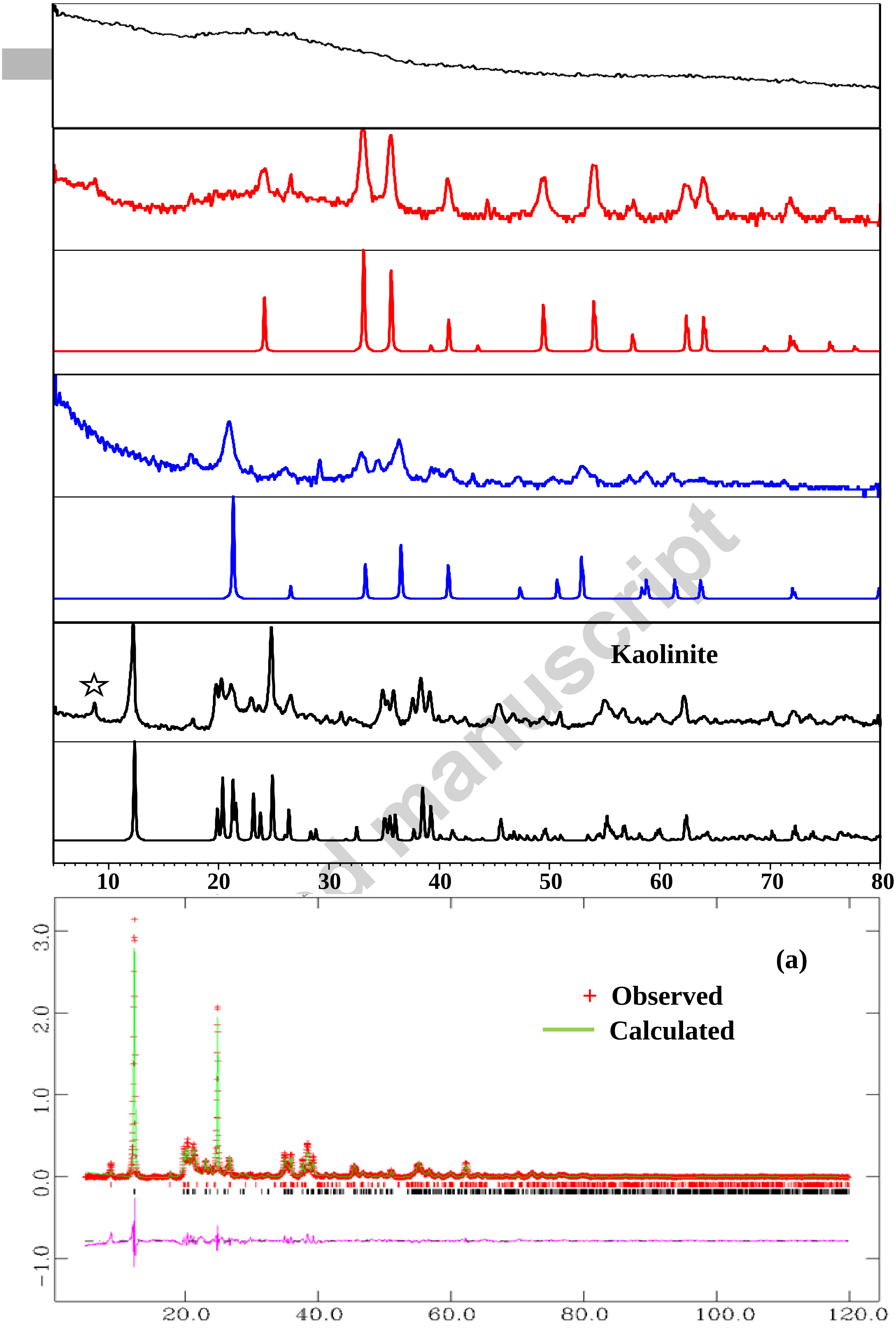


Fig 1. Powder XRD patterns of (a) calculated and observed pattern of kaolinite, (b) Kaolinite (Joint Committee on Powder Diffraction Standards (JCPDS) pdf number: ☆89-6538; - 2Θ = 10.09°-Iillite impurity),(c) Raw FeOOH (Goethite, JCPDS number: 03-0251), (d) KH (Hematite, JCPDS number: 24-0072) and (e) metakaolinite.

Figure 2

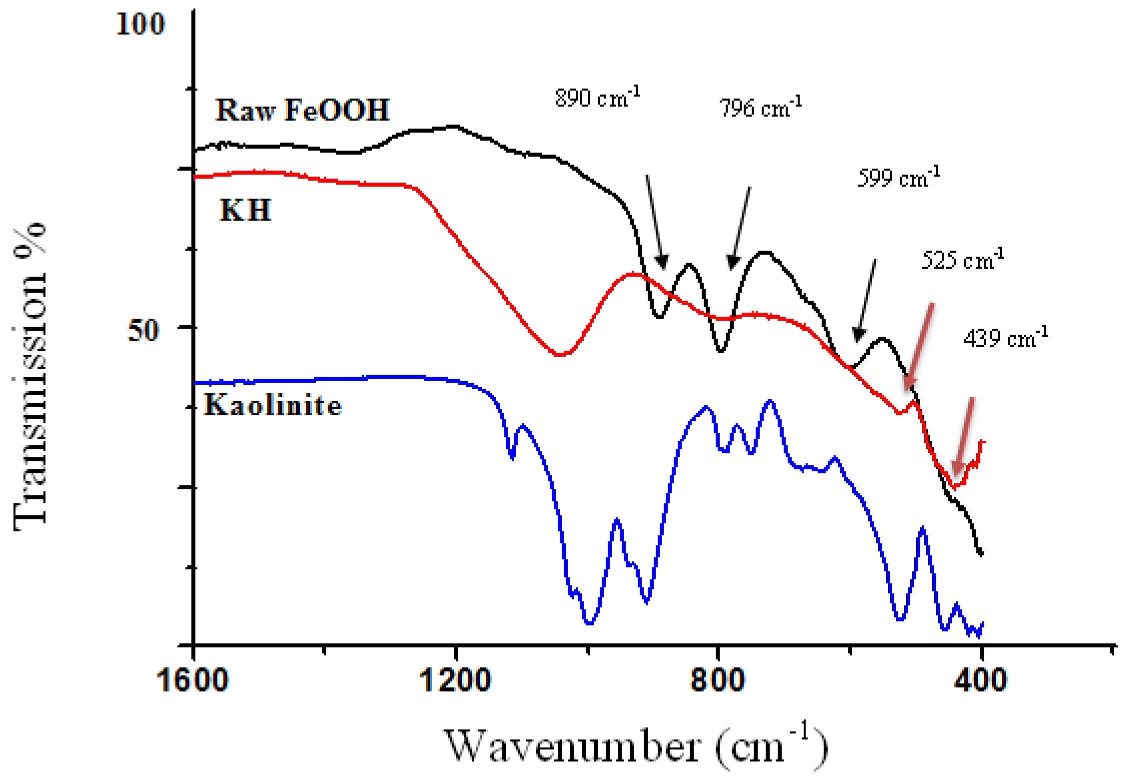


Fig 2. Attenuated total reflectance infrared spectrum of Kaolinite, Raw FeOOH (Goethite) and KH

Figure 3

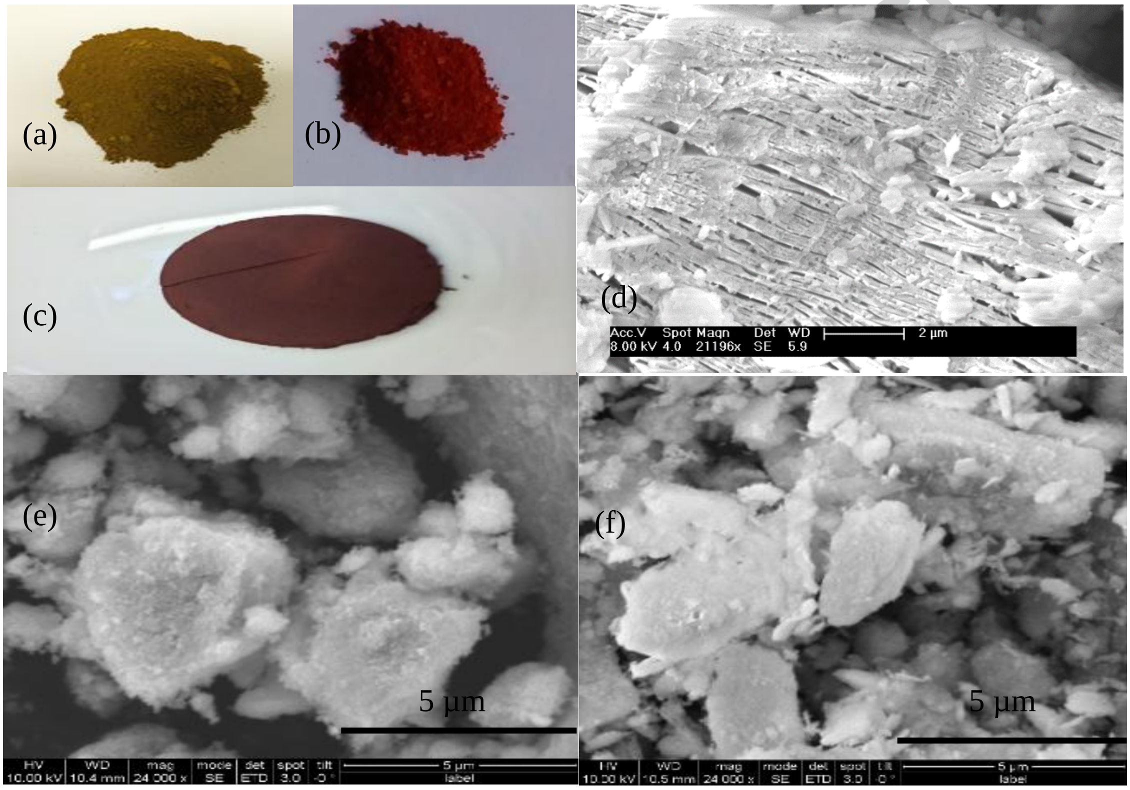
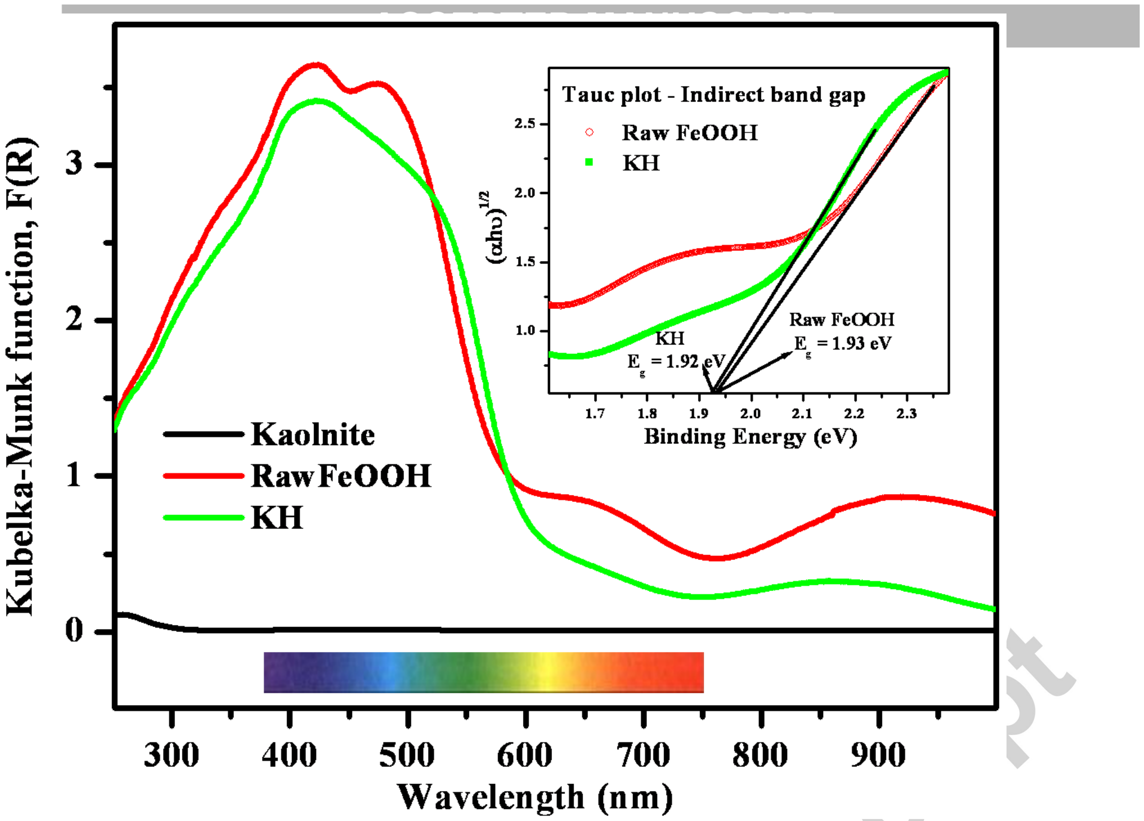
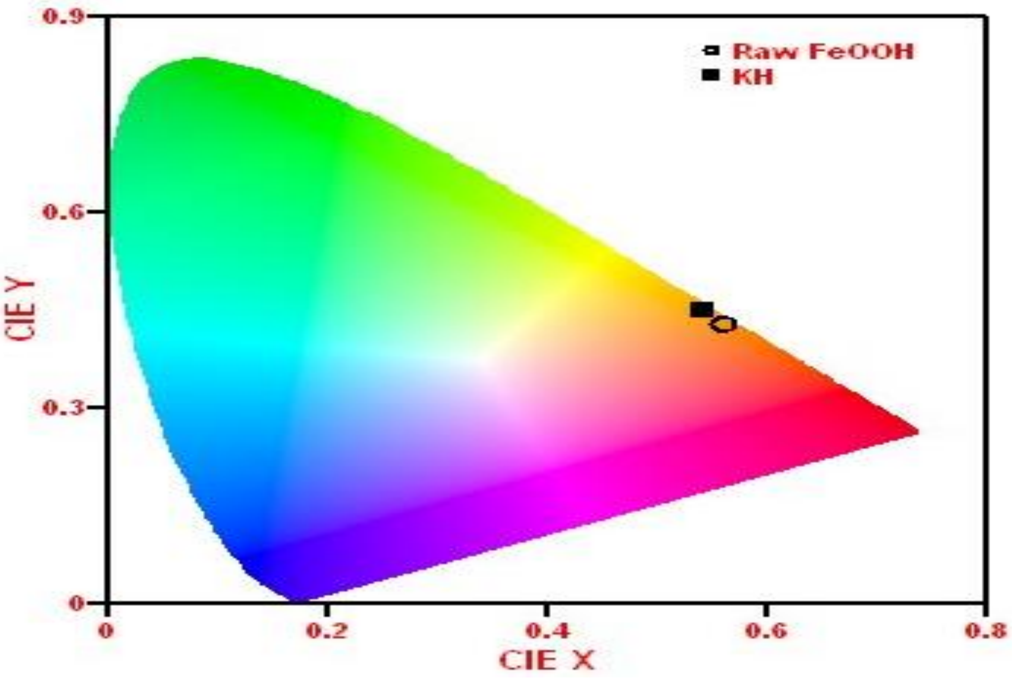


Fig 3. (a) Raw FeOOH (iron oxide sludge dried), (b) KH specimen, (C) alkali treated KH, (d) SEM image of kaolinite, (e) SEM image of Raw FeOOH and (f) SEM image of KH specimen

Figure 4



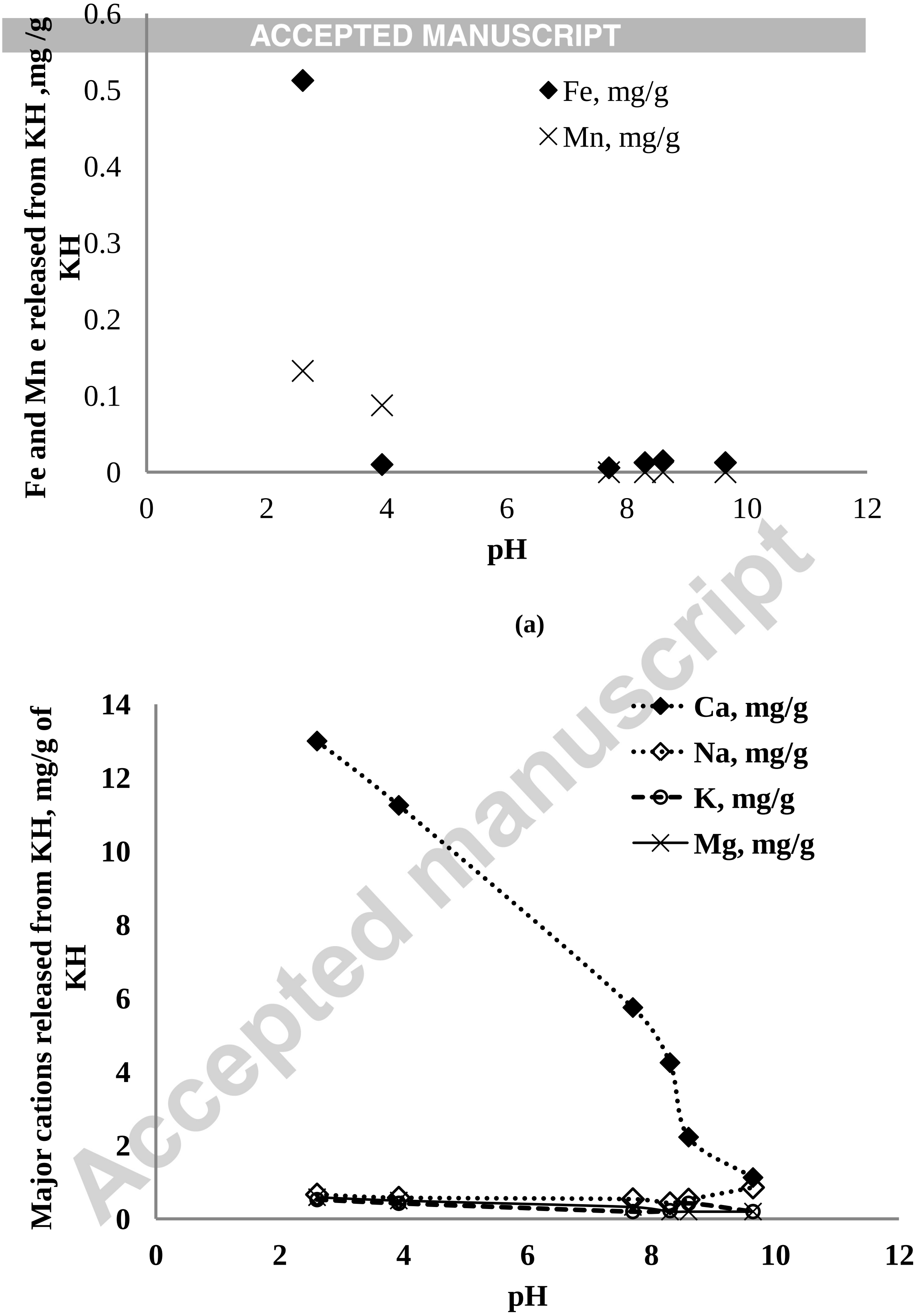
(a)



(b)

Fig 4. (a) Diffuse reflectance spectra of Raw FeOOH, KH and kaolnite samples; ((a) insert) Tauc plot for indirect band gap of Raw FeOOH and KH specimens and ; (b) CIE-Colour system of Raw FeOOH and KH.

Figure 5

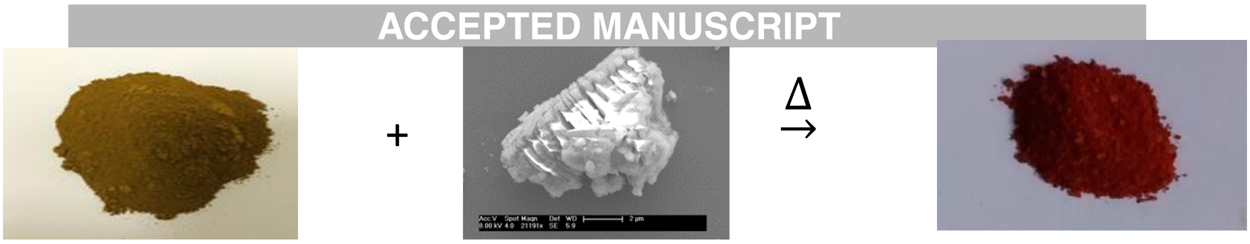

Fe and Mn e released from KH ,mg /g KH
0.6
0.5
0.4
0.3
0.2
0.1
0
0
2
4
6
8
10
12
pH
◆ Fe, mg/g
× Mn, mg/g
Major cations released from KH, mg/g of KH
14
12
10
8
6
4
2
0
0
2
4
6
8
10
12
pH
Ca, mg/g
Na, mg/g
K, mg/g
Mg, mg/g

(a)

(b)

Fig 5. (a) and (b) water leachable major cations

*Graphical Abstract



A kaolinite based red pigment was prepared using an ocherous iron oxide sludge recovered from an abandoned coal mine water treatment plant.

Highlights

- A red pigment was prepared by heating the kaolinite and the iron oxide sludge at kaolinite's dehydroxylation temperature.
- The colour properties of the iron oxide recovered from the coal mine water treatment system (Raw FeOOH) and the pigment.
- The pigment, the product of goethite-Kaolinite hydrothermal reaction can be a potential pigment for integrally coloured concrete.