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#### Journal of Water Process Engineering

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## Removal of fine iron-oxide particles after post-filtration in local potable water using an electrophoretic method



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#### ARTICLE INFO

# Article history: Received 3 August 2015 Received in revised form 4 January 2016 Accepted 10 January 2016 Available online 22 January 2016

Keywords: Electrophoretic deposition Fibre electrodes Limiting flux Iron oxide Zeta potential

#### ABSTRACT

Potable water from several residential areas on the east coast of Malaysia was filtered using a polyethersulphone (PES) membrane to separate the coarse and fine iron-oxide particles inside the pipelines. The as-received samples consisted of a wide distribution of particle sizes, ranging from 5  $\mu$ m to 400 nm. The concentration of fine iron-oxide particles inside a distribution system was extremely low. Hence, a specific method is necessary to concentrate and separate the fine particles from the coarse ones. To study the fine particles from the bulk, excess pressure was applied to the membrane filter so that the clogged particles were released into the permeate. A 100 kDa PES membrane was used to separate the particles, because the samples consisted of a wide molecular-weight cut-off range from 89 g/mol goethite ( $\alpha$ -FeOOH) to 231 g/mol hematite (Fe<sub>2</sub>O<sub>3</sub>). After the filtration process, the size distribution of permeated particles reduced to 550-400 nm. Through X-ray diffraction analysis, numerous polymorphs such as α-FeOOH, Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> and maghemite were detected from the samples. The zeta potential value of the permeated particles changed from -18.5 to  $-13\,\mathrm{mV}$ , suggesting that the dispersity of permeated iron-oxide particles became unstable, but remained adequate for electrophoretic deposition (EPD). The fibrous carbon electrode used in the EPD process, could remove up to 87% of the permeated iron-oxide particles compared to solid carbon electrodes (<56%). A high-surface-area, porous electrode and a moderate applied voltage were preferred in order to minimise gas formation, reduce the electro-osmosis effect and increase the deposition efficiency.

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#### 1. Introduction

Drinking water, or potable water, is defined as treated water that is used for domestic purposes such as drinking, cooking and personal hygiene. It is safe for consumption by humans with low risk of immediate or long-term harm. In tropical regions such as Malaysia, potable water is distributed using pipelines that are made from iron-based materials. During the distribution process, deterioration of the inner pipe interface occurs, owing to corrosion, contributing to the accumulation of iron-oxide particles in the bulk. At the initial stage of the corrosion process, the solid iron oxides exist in low-crystalline form, but over time, the compounds trans-

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form into higher crystalline forms, such as goethite ( $\alpha$ -FeOOH) or hematite ( $Fe_2O_3$ ). Dixit and Hering pointed out that the rate of these transformations depends on temperature, pH and the presence of other co-occurring solutes [1]. During the corrosion process, the morphology of the inner iron pipe surfaces consists of a porous core, a hard shell layer (HSL) and a surface layer. The porous core regions consist of enormous fine tubercles that are composed of  $\alpha$ -FeOOH, lepidcrocite ( $\gamma$ -FeOOH), green rusts, magnetite (Fe<sub>3</sub>O<sub>4</sub>), Fe<sub>2</sub>O<sub>3</sub>, ferrous hydroxide [Fe(OH)<sub>2</sub>], ferric hydroxide [Fe(OH)<sub>3</sub>] and siderite (FeCO<sub>3</sub>). The HSL lies on top of the core region and varies from one to a few millimeters in thickness [2], and it is typically composed of Fe<sub>3</sub>O<sub>4</sub> and  $\alpha$ -FeOOH [3,4]. Generally, the outermost or surface layer can be up to several millimeters thick. The layer is more heterogeneous and can contain  $\alpha$ -FeOOH,  $\gamma$ -FeOOH, Fe(OH)<sub>3</sub>, silicates, phosphates and carbonates [5]. Sarin et al. and Senftle et al. explained that, under ambient conditions, the corrosion process is

dominated by iron ionisation followed by solid reduction before the iron is released as a solid ferrous form [4,6]. Later, it could either be dispersed in the bulk or deposited as a scale on the pipe-wall surface [7,16]. The solid fragment of iron-oxide substances or tubercles that are released into the potable water initially exist in the milimeter-to-submicron size order. The coarse tubercles are easy to sediment, but the fine tubercles are well-dispersed and can flow homogeneously inside pipelines or distribution systems. Fine ironoxide particles that exist in the nanometer size order have a higher surface activity compared to coarse ones, and have the potential to form natural adsorbent particles. As a result, the fine tubercles that are exposed with a long residence time inside a distribution system will turn into coarse particles with the potential of promoting biofilm growth, microbial algae activities [3,8], green rust [9] and adsorption or accumulation of some contaminant such as arsenic [10] and radium [11].

The concentration of fine iron-oxide particles inside a distribution system is extremely low and difficult to study. In order to study the particles, a specific method is necessary to concentrate and separate the fine particles from the coarse ones. In the present study, a batch filtration process is used to increase the concentration of iron-oxide particles. The collected iron-oxide particles were filtered and trapped inside a membrane matrix. By increasing the applied pressure to slightly more than the limiting flux value, the fine iron-oxide particles that were trapped in the matrix were released to the permeate and could be measured and studied. The released iron-oxide particles were subjected to a removal process by means of electrophoretic deposition. According to Naim et al., electric-assisted separation or electrophoretic deposition (EPD) was conducted, because of its potential to be applied directly, without any additional treatment or chemicals [12]. However, before EPD could be applied, a preliminary study on the characterisation of permeated iron-oxide particles that were released to the permeate was necessary. This study also emphasised the potential of removing and depositing the permeated iron-oxide particles after applying the EPD system in terms of removal efficiency and deposit-substrate morphology. Characterisation of fine iron-oxide particles in the pre- and post-filtration steps is important in order to study the feasibility of applying EPD in the permeate region or after the filtration process. In this study, a particular concern was given to the permeation of iron-oxide particles and the surface charge of the iron-oxide particles after passing the membrane. The EPD process is preferable if the permeated iron-oxide particles have a high surface charge or adequate zeta potential value. If the surface charge is completely stripped off by the membrane matrix, an additional treatment might necessary to increase the surface charge of the particles. Particular concerns when studying the separation parameters, such as limiting flux and applied pressure, are taken into account so that losses of particle charges through concentration polarisation and friction within the membrane matrix can be minimised. At the end of this work, an alternative method to increase the concentration of fine iron oxides removed by EPD was also demonstrated by increasing the electrode surface area and porosity.

#### 2. Experimental

#### 2.1. Collection of samples

A volume of 10 L of potable water was taken from several residences in Kuantan, Pahang, Malaysia. The tap water was collected in plastic bottles and tightly closed to prevent interaction with the surrounding environment. The taps and the pipelines were estimated to be about 10 years old and have a diameter of 2.5 cm. Both the taps and the pipes were made from galvanised iron (BS1387:1967). The collected water samples were tested according

**Table 1**Analysis of the as-received samples according to the Malaysian Food Act, Water and Packaged Drinking Water Standard (Health) [28].

Parameter	Results, g/m <sup>3</sup>	Standard, g/m <sup>3</sup>
Colour	<2.5	15
Turbidity	1.5	5
pH	6.5	6.5-8.5
Aluminium, mg/L	Not detected (<0.06)	0.2
Arsenic, mg/L	Not detected (<0.0001)	0.05
Cadmium, mg/L	Not detected (<0.003)	0.005
Chromium, mg/L	Not detected (<0.03)	0.05
Copper, mg/L	0.05	1.0
Cyanide, mg/L	Not detected (<0.003)	0.1
Fluoride, mg/L	Not detected (<0.01)	1.5
Total hardness, mg/L	1.36	500
Iron, mg/L	0.05	0.3
Lead, mg/L	0.01	0.05
Magnesium, mg/L	0.16	150
Manganese, mg/L	Not detected (<0.002)	0.001
Mercury, mg/L	Not detected (<0.0001)	0.001
Mineral oil, mg/L	Not detected (<0.1)	0.3
Nitrate, mg/L	0.16	10
Phenol, mg/L	Not detected (<0.001)	0.002
Residual Chlorine, mg/L	Not detected (<0.1)	0.1
Selenium, mg/L	Not detected (<0.0001)	0.01
Silver, mg/L	Not detected (<0.005)	0.05
Sodium, mg/L	0.82	200
Sulfur, mg/L	Not detected (<0.01)	400
Zinc, mg/L	0.05	5

to the national standards for water quality (Malaysian standard), as shown in Table 1 [28].

#### 2.2. Filtration unit and membrane selection

A membrane made of polyethersulphone (PES) with an active surface area of 40 cm<sup>2</sup> was used as a filter for the filtration process. The filtration unit and the membrane system were purchased from Sartorius Stedim Malaysia Sdn. Bhd, Malaysia. The membrane had a 10 nm pore size with a 100 kDa molecular weight cut-off (WMCO). The process flow of membrane filtration from the source until the analysis stage is shown in Fig. 1.

#### 2.3. Concentration of iron-oxide particles

The concentration of iron-oxide particles for samples asreceived, permeated and remaining after the deposition was measured using ICP-OES (iCAP6000, Thermo Scientific, USA). The removal percentage calculation was performed to identify the EPD efficiency. The inductively coupled plasma was used to identify the metal element. The nitric acid digestion method was performed to reduce the particulates in order to form free ions.

#### 2.4. X-ray diffraction (XRD) analysis

Each batch of the water samples was pre-filtered using a  $0.2~\mu m$  polytetrafluoroethylene (PTFE) syringe filter. The purpose of pre-filtration is to avoid uncontrolled clogging, owing to the very wide particle size distribution. The process also removes unwanted contaminants such as debris and coarse natural organic matter (NOM), so that membrane filter clog can be minimised [13]. The thin film of membrane with accumulated particles was calcined in the furnace at  $600~^{\circ}\text{C}$  for 5 hours, and then preserved in the furnace for several hours in order to promote the crystallisation process [14]. The calcinated powders were analysed using an X-ray diffractometer (Model APD 2000, G.N.R., Italy). A copper anode was used as a radiation source (CuK $\alpha$ ) with a wavelength of 1.54 nm. A voltage of 40 kV and current of 30 mA were used in XRD analysis [13]. The XRD analysis was perfor ed with scanning angles between 2 and  $80^{\circ}$ 

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