

# 11 Å tobermorite from cement bypass dust and waste container glass: A feasibility study

N.J. Coleman\*, C.J. Trice, J.W. Nicholson

School of Science, University of Greenwich, Chatham Maritime, Kent, ME4 4TB, UK

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## ABSTRACT

A novel one-step hydrothermal synthesis of 11 Å tobermorite, a cation exchanger, from a unique combination of waste materials is reported. 11 Å tobermorite was prepared from stoichiometric quantities of cement bypass dust and waste container glass at 100 °C in water. The product also comprised 10 wt.% calcite and trace quartz as residual parent phases from the cement bypass dust. In a batch sorption study at 20 °C the uptakes of Cd<sup>2+</sup> and Pb<sup>2+</sup> by the waste-derived tobermorite product were found to be 171 mg g<sup>-1</sup> and 467 mg g<sup>-1</sup>, respectively, and in both cases the removal process could be described using a simple pseudo-second-order rate model ( $k_2 = 2.30 \times 10^{-5} \text{ g mg}^{-1} \text{ min}^{-1}$  and  $5.09 \times 10^{-5} \text{ g mg}^{-1} \text{ min}^{-1}$ , respectively). The sorption characteristics of the 11 Å tobermorite are compared with those of other waste-derived sorbents and potential applications are discussed.

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## 1. Introduction

Tobermorites are naturally occurring layered calcium silicate hydrate minerals which comprise a double Ca–O sheet bound on both sides by anionic wollastonite-like silicate chains running parallel to the b-axis direction as indicated in Fig. 1 (Merlino et al., 1999). The inter-layer region accommodates labile charge-balancing cations and water molecules. Members of this family are characterised in terms of the length of their c-axis, which is dependent upon the number of water molecules per unit formula. There are three principal polytypes of this mineral, viz. 14, 11 and 9 Å tobermorite, whose names relate to the approximate d-spacings of their (0 0 2) Bragg reflections (i.e. their basal spacings).

Phase pure 11 Å tobermorite, whose ideal composition is Ca<sub>5</sub>Si<sub>6</sub>O<sub>18</sub>H<sub>2</sub>–4H<sub>2</sub>O, and its Al- and Fe-substituted counterparts have been synthesised under alkaline hydrothermal conditions at temperatures between 80 and 225 °C and their ion exchange properties have been investigated with respect to their application in nuclear and hazardous wastewater benefaction (Komarneni et al., 1982, 1988; Komarneni and Roy, 1985; Mitsuda and Taylor, 1975; Mostafa et al., 2009; Siauciunas et al., 2004). To date the syntheses of 11 Å tobermorites reported in the literature have generally been carried out using analytical grade reagents such as sodium silicate and fumed silica or natural zeolites and clays; however, recent efforts have been made to replace laboratory grade feedstock constituents with by-products such as municipal waste incineration fly ashes (Yao et al., 1999), oil shale ash (Reinik et al., 2007) and newsprint recycling residue

(Coleman, 2005, 2006). In this paper the authors explore the feasibility of using a unique combination of two high-volume waste materials – cement bypass dust (CBPD) and discarded soda–lime–silica glass containers (SLSG) – for the one-step hydrothermal synthesis of 11 Å tobermorite.

CBPD (also known as cement kiln dust) is a by-product of the manufacture of Portland cement which is of little market value and is principally consigned to landfill where it poses a risk of groundwater contamination (Siddique, 2006). It is generated as dust particles which are collected from exhaust gases by electrostatic precipitators during the high-temperature calcination process. A small proportion of CBPD may be returned to the kiln as feedstock although high concentrations of alkali metals and sulphates largely prohibit its direct reuse in cement manufacture. Compositional variability further restricts its potential to be used elsewhere as a secondary raw material, although current research indicates that it may find limited application in the production of asphalt concrete and controlled low-strength construction materials (Siddique, 2006).

Discarded SLSG (also known as cullet) constitutes a significant component of the municipal solid waste stream in every developed nation. The recycling of container glass (jars, bottles etc.) is both an environmental concern and a political obligation within the European Union; however, the recycling potential of SLSG is frustrated by a range of political, aesthetic and technical obstacles. Poor collection infrastructure and colour mismatch limit the supply of suitable cullet that can be effectively recycled into new containers. Furthermore, large quantities of recycled cullet alter the well-defined nature of the feedstock during glass manufacture causing variations in composition and redox potential which compromise the quality of the finished articles (Karamberi and Moutsatsou, 2005). In response to this problem, various research projects are presently underway to identify

\* Corresponding author. Tel.: +44 7908 017088; fax: +44 20 8331 9805.

E-mail address: [nj\\_coleman@yahoo.co.uk](mailto:nj_coleman@yahoo.co.uk) (N.J. Coleman).

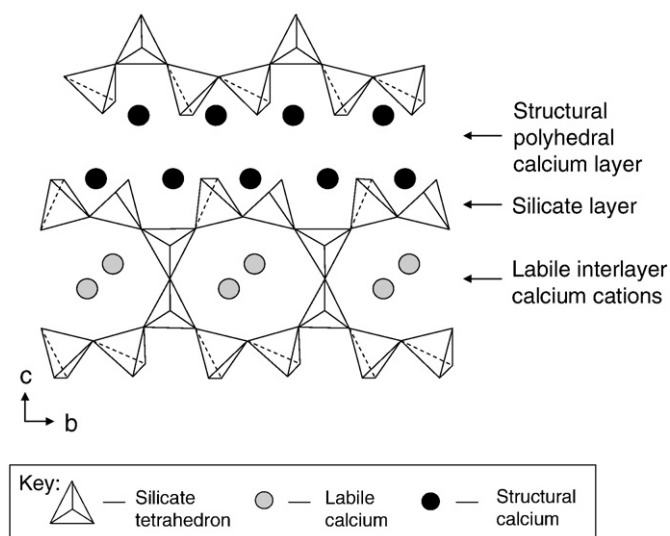


Fig. 1. A projection of 11 Å tobermorite in the *bc*-plane.

alternative markets for waste cullet as a secondary feedstock in the production of materials such as aggregates, concretes, ceramics and abrasives (Karamberi and Moutsatsou, 2005; Matteucci et al., 2002; Park et al., 2004).

In view of the necessity to conserve natural mineral resources and to reduce the volume of industrial waste that is destined for landfill, an investigation to examine the feasibility of preparing 11 Å tobermorite from an exclusive combination of CBPD and SLSG has been carried out. Kinetic batch sorption data for the uptake of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  ions by the CBPD/SLSG-derived 11 Å tobermorite product are also reported and potential applications are discussed.

## 2. Materials and methods

### 2.1. Hydrothermal synthesis and product characterisation

CBPD arising from the manufacture of ordinary Portland cement was supplied by CEMEX UK Operations Ltd. and used 'as received' with no further preparation. SLSG containers were recovered from the municipal waste stream in Rochester, Kent, UK and ground in an agate TEMA mill to pass 125  $\mu\text{m}$  prior to use. Oxide analyses for CBPD and SLSG were obtained by X-ray fluorescence spectroscopy (XRF) (Materials Research Institute, Sheffield Hallam University, Sheffield, UK) and are presented in Table 1. The major crystalline phases of CBPD were determined by powder X-ray diffraction analysis (XRD) using a Siemens Kristalloflex 810 X-ray diffractometer and  $\text{CuK}\alpha$  source.

11 Å tobermorite samples were prepared by dry-blending 3.0 g CBPD and 2.7 g SLSG for 5 min prior to the addition of water and hydrothermal treatment. Optimum yields of tobermorite are generally obtained when the reaction composition falls within the following molar ratios:  $0.80 < \text{Ca}/$

$[\text{Si} + \text{Al} + \text{Fe}] < 0.85$  and  $0.00 < [\text{Al} + \text{Fe}]/[\text{Si} + \text{Al} + \text{Fe}] < 0.17$ ; hence, these mix proportions were selected to achieve target ratios of 0.84 and 0.10, respectively (Coleman, 2005). Syntheses were carried out in duplicate at a constant solid:solution ratio of  $0.095 \text{ g cm}^{-3}$  by heating the reaction mixtures under sessile conditions at 100 °C in hermetically-sealed PTFE vessels under autogenous pressure for 2, 7 and 14 days (as summarised in Table 2). In each case, the charge was recovered by gravitational filtration, washed with deionised water to  $\text{pH} \sim 8$ , dried to constant mass in air at 40 °C and stored in air-tight polypropylene containers until required.

Reaction products, Tob-2, Tob-7 and Tob-14 (as defined in Table 2), were characterised by powder XRD and by simultaneous differential thermal analysis and thermo-gravimetry (DTA-TG) using a Stanton Redcroft 673-4 Differential Thermal Analyser with a calcined alumina reference. Samples were heated at a rate of  $20 \text{ }^\circ\text{C min}^{-1}$  to a maximum temperature of 1000 °C.

### 2.2. Sorption of $\text{Cd}^{2+}$ and $\text{Pb}^{2+}$ by Tob-14

The kinetics of removal of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  by Tob-14 were determined by single metal, sessile, batch sorption experiments at 20 °C. In each case 50 mg of Tob-14 were contacted with  $0.2 \text{ dm}^3$  metal nitrate solution at an approximate metal concentration of 0.5 mM in screw-capped polypropylene bottles. The residence times for specimens were 3, 6, 24, 72 and 168 h, after which, the supernatant liquors were recovered by centrifugation at 3000 rpm and their pH recorded. Each experiment was carried out in duplicate and the recovered solutions were appropriately diluted and analysed by optical emission inductively coupled plasma spectrophotometry (ICP-OES) using matrix-matched standards. In each case, the relative standard deviations of the mean metal-uptake data were lower than 6%. The kinetic data for the removal of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  by Tob-14 were then fitted to pseudo-rate models (as outlined in the following section) by linear regression analysis. The recovered solids were dried to constant mass at 40 °C in air and re-analysed by powder XRD.

## 3. Kinetic models

The pseudo-first-order rate model which describes the sorption of a solute on to a solid surface can be expressed in the following way (Coleman et al., 2006a; Wu et al., 2001):

$$\frac{dq_t}{dt} = k_1(q_e - q_t)$$

where  $k_1$  is the apparent pseudo-first-order rate constant (in  $\text{min}^{-1}$ ),  $q_t$  is the extent of sorption at time  $t$  (in  $\text{mg g}^{-1}$ ), and  $q_e$  is the extent of sorption at equilibrium (in  $\text{mg g}^{-1}$ ). This pseudo-rate law is used to describe processes in which the reaction rate,  $dq_t/dt$ , is proportional to the number of available sorption sites,  $(q_e - q_t)$ . The linear, integrated form of this equation for the boundary conditions;  $q_t = 0$  at  $t = 0$  and  $q_t = q_t$  at  $t = t$ , can be written as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

Hence, the pseudo-first-order rate equation is obeyed when a linear relationship exists between  $\log(q_e - q_t)$  and  $t$ , in which case  $k_1$  may be estimated from the gradient of the plot.

Table 1  
Compositions of cement bypass dust and soda-lime-silica container glass.

Major oxide composition (wt.%)								
CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	L.O.I. <sup>a</sup>
<i>Cement bypass dust</i>								
58.81	14.37	4.85	2.19	1.12	1.39	1.89	9.05	3.21
<i>Soda-lime-silica glass</i>								
11.11	72.50	1.31	0.10	0.96	13.22	0.44	0.18	–

<sup>a</sup> Loss on ignition.

Table 2  
Reaction conditions and mixture compositions.

Sample	Reaction conditions	Ca:Si:Al:Fe:H <sub>2</sub> O molar ratios
Tob-2	100 °C, 2 days	0.93: 1.00: 0.09: 0.02: 84
Tob-7	100 °C, 7 days	0.93: 1.00: 0.09: 0.02: 84
Tob-14	100 °C, 14 days	0.93: 1.00: 0.09: 0.02: 84

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