



Research Article

Use of carbon coated ceramic barriers for adsorptive removal of fluoride and permanent immobilization of the spent adsorbent barriers

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Abstract

This work is focused on the coating of carbon particles on a clay based flat ceramic substrate by vacuum driven filtration deposition and its use for the separation of fluoride from contaminated water. A thin and smooth layer of nano-aluminum oxyhydroxide deposited carbon over the porous clay based flat ceramic support was obtained by the simple vacuum suction technique. The coated membrane was able to remove fluoride with good efficiency. Post adsorption barriers containing toxic fluoride ions cannot be simply thrown away but fluoride ions need to be immobilized in it to prevent its leaching from it. Therefore, another important component of the work is the permanent immobilization of fluoride adsorbed membrane (FCM) in cement clinkers by reacting with lime and clay by black meal method of clinker preparation. All the necessary phases like tricalcium silicate ($3\text{CaO}\cdot\text{SiO}_2$), dicalcium silicate ($2\text{CaO}\cdot\text{SiO}_2$), tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) and tetra calcium aluminoferrite ($4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$) found in a typical portland cement clinkers are observed in the prepared clinkers and confirmed from X-ray diffractometry. The studies showed that conventional firing temperature of 1450 °C for clinker formation could be lowered to 1350 °C by the addition of fluoride adsorbed ceramic waste due taking advantage of mineralizers such as F^- and Al^{3+} and the carbonaceous materials which facilitated the firing process. The fluoride leaching studies confirmed the acceptability of the process.

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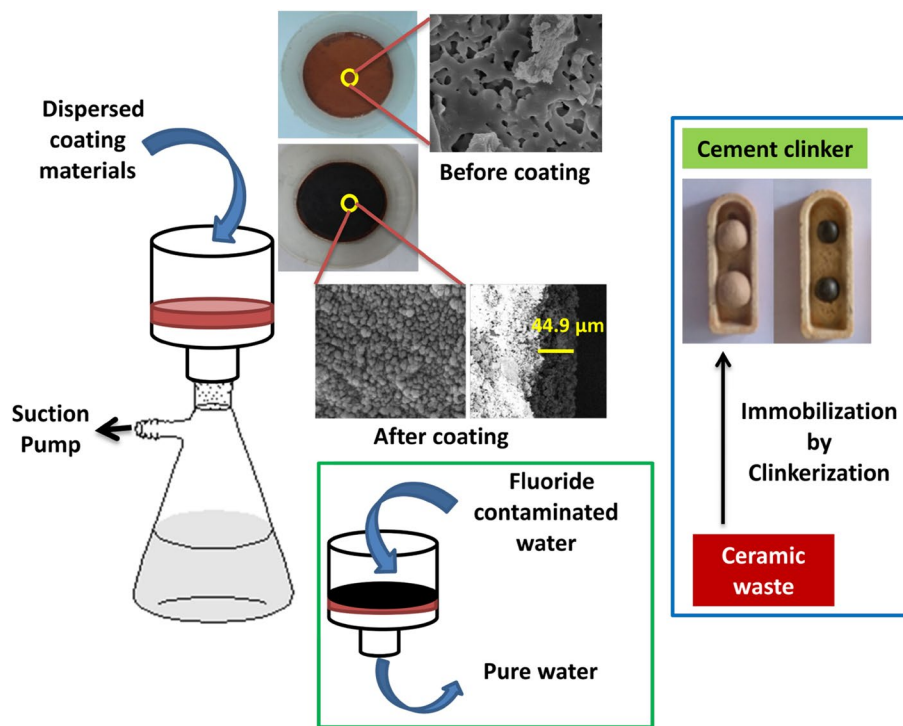


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Graphical abstract



Keywords Carbon coated ceramic membrane · Defluoridation · Permanent immobilization · Cement clinker

1 Introduction

Development of ceramic filters and membranes for water purification is known for years. Due to their distinct advantages over other filtration barriers or adsorbent materials for water treatment, ceramic membranes and barriers are gaining very high attention in recent years [2, 11]. Ceramic filtration barriers are often composed of certain macroporous support layers over which smoother coats of functional materials are made to obtain asymmetric membranes. Different methods such as chemical vapour deposition (CVD), spin and dip coating and vacuum or pressure filtration are commonly used for such purposes. Out of these coating methods vacuum or pressure filtration method has the advantage of easy operability and which can be applied for large area coating [9]. Carbonaceous materials are commonly deposited over such supports for applications such as enhanced pollutant separation, improved catalyst design, water recovery and other related applications [2]. The uniform coats over the porous filters are impermeable for the colloidal particles but permeable for the liquid. The thickness and pore size of the coating layer is dependent on the extent of the applied force and

the nature of the colloidal slip used for coating [13, 15]. Fabrication of membrane by vacuum filtration methods is reported by many researchers, which includes deposition of hydrotalcite thin film [9] and carbonaceous materials on alumina ceramic surface for many applications including water purification [5–7, 16, 23]. For coating of membranes by vacuum filtration deposition method, a low viscosity slip of good dispersal stability is required to avoid bridging among particles, which otherwise lead to pore blocking of the supports [13].

Fluoride is one of the most toxic inorganic pollutants which are reported to cause a number of ill effects to human beings like fluorosis, arthritis, hip fractures, infertility and osteoporosis [3, 20, 27] when consumed in excessive level beyond the permissible limit of 1.5 mg L⁻¹ [26]. To control such difficult water pollutants commonly adsorption over economic adsorbent surfaces is applied. Most of the adsorbents show very good adsorption but can be used for one time only. In such cases handling of contaminant loaded materials produced after adsorption is another major environmental concern [4, 12]. Permanent immobilization of such waste materials has been studied by several researchers [8, 10, 20–22, 24]. Mainly,

under the 3R (Reduce, Reuse and Recycle) dictum of modern circular economy use of such materials for preparation of valuable materials would be important from the sustainability aspect and side by side leading to cost offsetting in water treatment processes.

Thus in this study, in total we report the preparation of carbon coated ceramic membrane by a simple vacuum-assisted slurry deposition method to obtain dense and smooth surfaces over porous flat clay based substrates. The coated membranes were used for the separation of fluoride from contaminated water. The study also reports a method for immobilization of fluoride adsorbed ceramic barriers in a lime silica matrix. Post defluoridation ceramic barriers were used as a partial replacement for clay in clinker preparation and it was found that phases required for clinker formation can be obtained by reacting the fluoride waste with lime and clay in a calculated ratio thereby raising a scope of utilization of such waste material with a prospective shift from linear economic position to a more sought after circular economic position [8, 10].

2 Experimental section

2.1 Chemicals and materials

All chemical reagents used were of analytical grade (AR). Sodium hydroxide (NaOH), Hydrochloric acid (HCl) and Nitric acid (HNO_3) were purchased from HIMEDIA (India). 100 mg g^{-1} fluoride stock solutions and sodium fluoride (NaF) salt was purchased from Merck (Germany). Ammonia (NH_3) was obtained from Nice Chemicals (India) and Aluminium chloride hexahydrate ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) was purchased from Otto Kemi (India).

2.2 Preparation of coating materials

Collection of *Ipomoea carnea* commonly known as pink morning glory stems and its carbonization is reported in our previous work [19]. Briefly, to obtain good quality low temperature carbonized carbon, Sun dried *Ipomoea* stems were carbonized in a muffle furnace (IKON, India, model no-Ik-108, size 6" × 6" × 12", Rating 4 kW) at an optimized temperature of 450 °C for 1 h. Carbonized stems were washed with double distilled water and dried in a hot air oven at 40 °C overnight and finally ground in a planetary micro pulverizer (M/S Fritsch, Germany) at 600 rpm for 2 min using zirconium balls.

Preparation of nano-aluminum oxyhydroxide deposited carbon (NAOC) is reported in our previous work [19]. Briefly, well ground carbons was functionalized by 5 N HNO_3 solutions at 80 °C for 1 h under refluxed condition and then filtered, washed and dried in a hot air oven at

110 °C until it was completely dried. The dried activated carbons were further modified with aluminum oxyhydroxide nanoparticles. For this purpose, 2 g activated carbon was dispersed in 200 mL distilled water at pH 10.5. $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ salt was added in 1:5 ratios to the dispersion and the mixture was stirred for 3 h. The mixture was filtered and washed with double distilled water to make the pH neutral. The residue cake thus obtained was dried in a hot air oven at 40 °C overnight. It was then ground well and used for deposition over ceramic preform.

2.3 Preparation of ceramic support

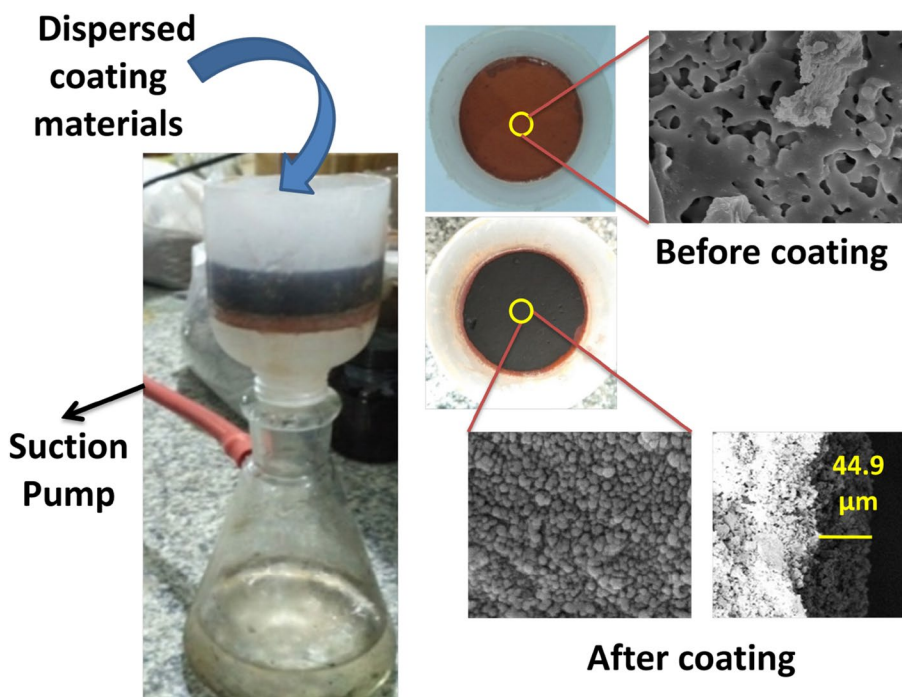
Preparation of clay based flat ceramic substrates is reported in some of our previous works [18]. Briefly, a 3:5:4 ratios of finely ground and sized Potters' clay (DKC) obtained from Dhekial region of Assam, India, Stone Quarry dust (SQD) supplied by local suppliers, and Tea dust (TD) obtained from local tea industry was taken and prepared a paste by adding 10 mL water to 45 g of the raw mixture. The paste of the mixed compositions was then moulded into dish shape using a hollow cylinder by wet pressing method. The prepared substrates was then dried at 40 °C for 24 h in a hot air oven and finally burnt at 1000 °C for 6 h in a muffle furnace to obtain the ceramic substrate.

2.4 Coating of modified carbon over ceramic support

To smoothening and covering the cracks and micro defects, the membrane support was coated with fine modified carbon to obtain a thin film over it. Another aim of this treatment is to enhance the pollutant removal capacity of the membrane. The coating of carbon particles over the ceramic support was carried out by vacuum-assisted film deposition method [9]. Prior to the treatment, the surface of the support disks was polished with sand paper and cleaned with distilled water in an ultrasonic bath to remove the loose particles and dried at 100 °C in a hot air oven for 6 h. For preparation of membrane thin film, 100 mL 0.1% and 0.05% colloidal aqueous dispersion of modified carbon was prepared under ultra sonication for 10 min.

A specific amount of dispersed colloidal solutions was poured from top of the support and allowed to pass through the membrane by using a vacuum system operated at a mild pressure of 50 kPa using a mechanical pump (Fig. 1). The colloidal particles were strongly adsorbed on the support and eventually distributed throughout the ceramic support. The coated film was dried at 100 °C in a hot air oven for 6 h.

Fig. 1 Schematic illustration of coating of ceramic substrate by vacuum assisted slurry deposition methods



2.5 Characterization of powdered coating materials and coated ceramic membrane

The coating materials and ceramic substrate before and after coating were characterized by different analytical methods and using analytical instruments. The surface morphology of the ceramic membrane was studied by FESEM (Field emission scanning electron microscopy) and EDS (Energy dispersive spectroscopy) techniques (Carl Zeiss (Sigma VP) instrument). The samples were dried in an oven (at around 100 °C) and kept in a vacuum desiccator for enough time before SEM analyses. FTIR (Fourier transform infrared) spectroscopy (model-IR Affinity-1) was used to study the surface functional groups by using KBr pellets in the range of 400–4000 cm^{-1} at the spectral resolution of 4 cm^{-1} , 40 scanned. Thermal stability of the samples (TGA/DTG) was conducted in a TG-DTA Apparatus (SDT Q 600, M/S TA Instruments, USA) using alumina as a reference at a heating rate of 20 °C per min under argon atmosphere at the flow rate of 100 mg L^{-1} . X-ray diffractometry (XRD) technique was used to observe the necessary phases formed in the samples by using X-ray powder diffractometer (Model Rigaku-Ultima IV, Japan) with the start angle 2°, stop angle 90° with a scanning rate of 2° per min and the target was CuK_α ($\lambda = 1.54056 \text{ \AA}$). The different phases were identified by comparing with standard ICDD (International Centre for Diffraction Data) card. The textural properties of the adsorbent (specific area) were tested by using an AutosorbiQ Station 1, equipped with an automated surface area at 77.35 K using Brunauet–Emmett–Teller (BET)

calculation for surface area. Dispersability of the coating materials was studied by jar test. For this 0.1 g of the samples were dispersed in 100 mL distilled water in a cylindrical jar of comparable height and radius and kept undisturbed for 7 days and monitored time to time.

2.6 Removal of fluoride by the coated ceramic barrier

A filtration unit was used for separation of fluoride contaminated water. The filtration unit was made by fixing a membrane (height = 0.4 cm, diameter = 4.2 cm) in a polyethylene cylindrical container (height = 3.7 cm, diameter = 4.3 cm). Synthetic fluoride solution of known concentrations was allowed to flow through the membrane under gravity and permeates was collected from time to time.

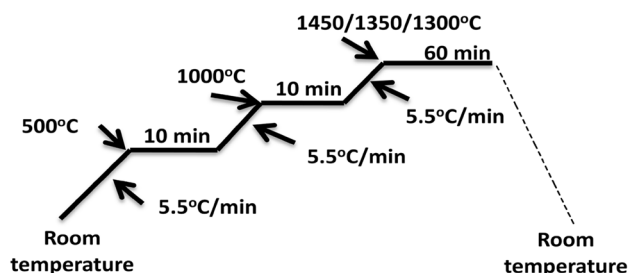


Fig. 2 Controlled burning of the raw nodules to obtain cement clinkers

Fluoride concentration was measured by an ion sensitive electrode (Orion 4 star, pH/ISE). The fluoride sensitive electrode was calibrated by using 0.1, 1, 10 mg L⁻¹ fluoride solutions. As the electrode does not measure fluoride complexed with silicon, iron, aluminium and other polyvalent cations. Total ionic strength adjustment buffer (TISAB-III) was added to the leachate solutions to destroy such complexes [17].

2.7 Clinkerization of ceramic waste with lime

The post defluoridation ceramic barriers were used as an partial replacement of clay used for cement clinker preparation. For clinker preparation commercial grade lime, Potters' clay (DKC) and ceramic wastes (CW) was mixed in a previously calculated ratio (Table. S.1) keeping in view of the all required parameters as practiced in the black meal process of cement manufacturing and ground thoroughly into fine powder in an agate mortar followed by milling in planetary ball mill. The clinkerization of the prepared pellets (made by hand driven pelletizer) was carried out by burning in a controlled manner in a digital muffle furnace at two different temperatures i.e. 1450 °C and 1350 °C separately for one h (Fig. 2). Cooling of burnt clinkers were carried out over a period of four h in air atmosphere inside the furnace.

2.8 Fluoride leachability study of the cement clinker

To study the possibility of the fluoride leaching from the cement clinker, a leachate solution from the clinkers was prepared following previously reported method by warming 1 g of the each cement clinker sample in 50 mL of doubled distilled water at 90 °C for 2 h followed by filtering and volume made up to 100 mL [17, 21]. The fluoride

content in the leachate was determined by using a fluoride ion-selective electrode as mentioned in Sect. 2.6.

3 Results and discussion

3.1 Characterization of the coating materials

Two types of coating materials were used for the coating purposes, the powdered form of the carbon obtained after carbonization of *Ipomoea* stems (RC) and the NAOC. The quality of coating of the coated ceramic barrier depends on the property of carbon to a large extent. Some important parameters such as dispersibility in water, zeta potential, BET surface area and surface morphology of the coating materials was studies and presented in Table S.2 and Fig. 3.

Zeta potential values showed that the surface of RC sample is negatively charged whereas in case of NAOC a positively charged surface was observed. Zeta potential values also reveals that the RC (-43.6 mV) sample is expected to be highly dispersible as compared to the NAOC (16.02 mV) sample due to higher zeta potential value. From the jar test carried out to check the dispersibility of the coating materials in water, is observed that RC is more stable as compared to NAOC.

BET surface area analysis showed that RC (3.351 m² g⁻¹) sample has very small surface area due to its planar structural morphology as also shown in FESEM images (Fig. 3) however it's surface area increased to 69.095 m² g⁻¹ after modification with nano aluminum oxyhydroxide. FESEM images show that the carbon surface has planar and regular sheet like morphology and in case of NAOC, spherical nanoparticles of size ≈ 50 nm are clearly observed over the planar sheets of RC.

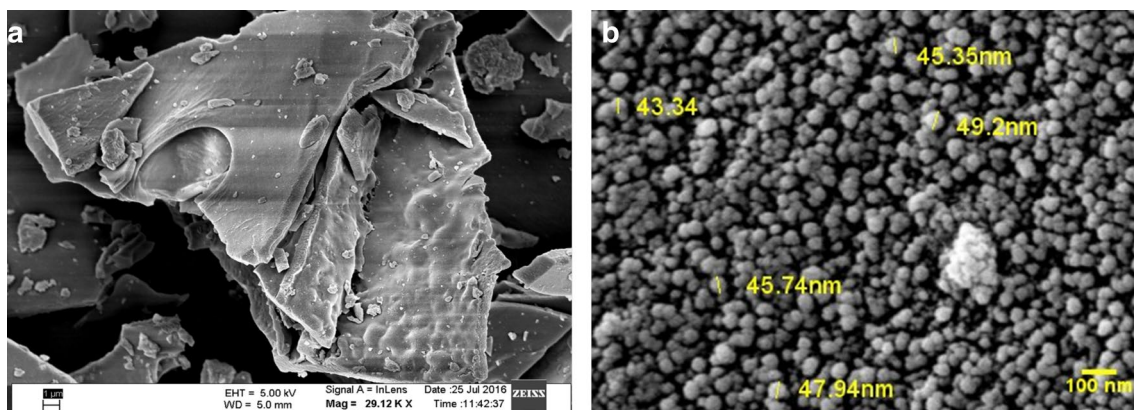


Fig. 3 FESEM images of **a** RC and **b** NAOC

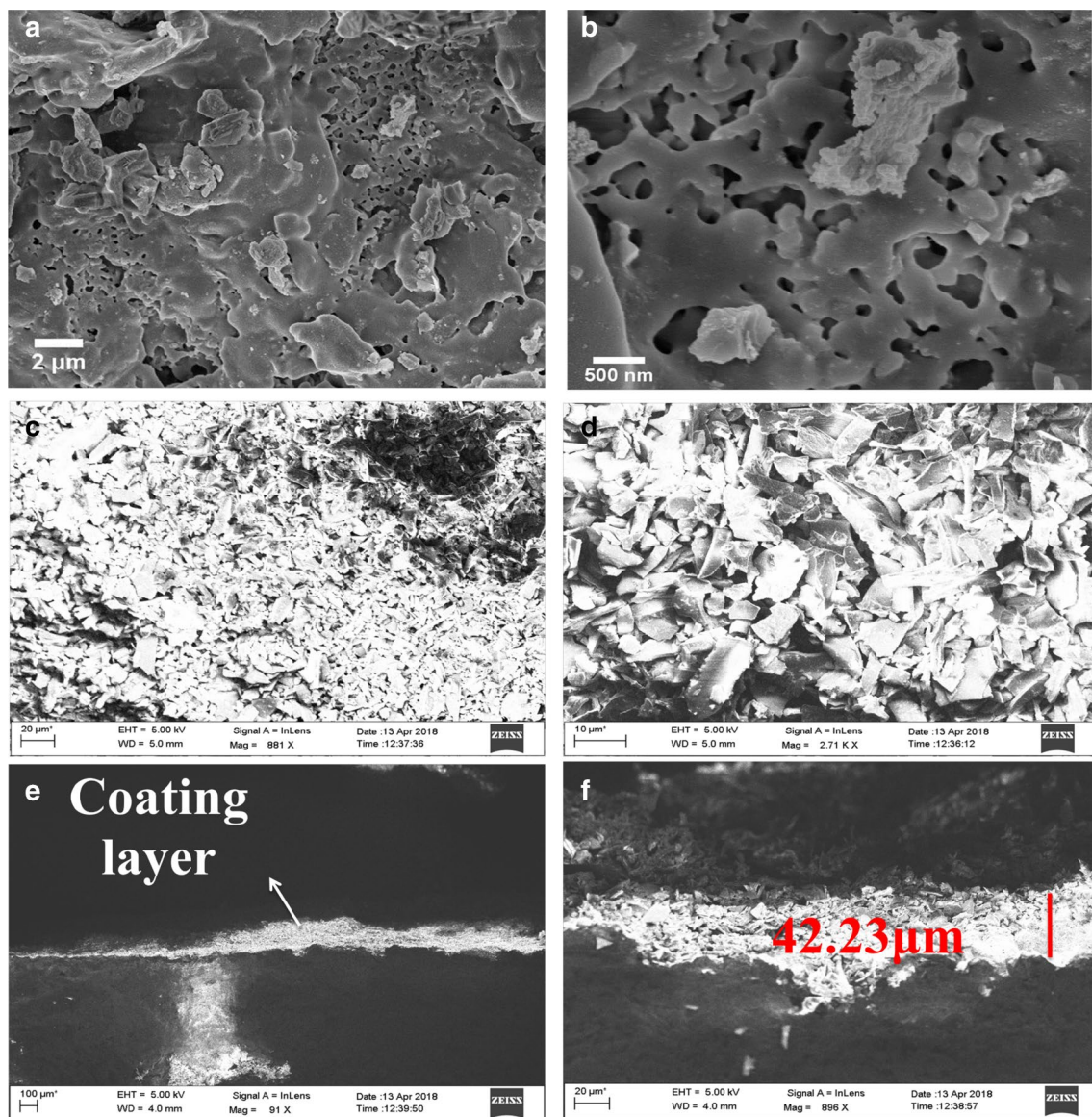


Fig. 4 FESEM images of **a, b** top view of ceramic barrier before coating, **c, d** top view of ceramic barrier after coated with RC sample and **e, f** cross section of coated barrier

3.2 Characterization of the coated barrier

For the coating purpose we have used clay based ceramic support prepared from kaolinite based potters' clay and stone quarry dust [22] with good chemical, thermal and mechanical resistance and highly porous with eventual distribution of similar sized pores (Fig. 4a, b). Some important properties of the ceramic substrate are tabulated in Table S.3. After coating of RC and NAOC by vacuum assisted deposition method, FESEM analysis was carried out to view the carbon layer on the ceramic substrate. FESEM images of the RC coated ceramic barriers are shown in Fig. 4. It is clear that the uncoated barrier surface is a rough and highly porous (Fig. 4a, b). However the

roughness of the uncoated barrier decreases on coating with RC slurry and a symmetric and dense deposition of flaky carbon particles on the barrier surface are observed (Fig. 4c, d).

From the FESEM images of the side view of the coated barrier, a distinct uniform layer of thickness $\approx 42.23 \mu\text{m}$ is observed which confirms the uniform coating of the carbon particles on the ceramic support (Fig. 4e, f). Energy dispersive spectroscopy (EDS) of different portion of the RC coated barrier is shown in Fig. 5. From Fig. 5a, b, the elemental analysis shows that 75.61 wt% is composed of carbon with 22.80 wt% oxygen and only 0.49 wt% Si. On the other hand, Si content increases to 26.33 wt% and O content increased to 54.43 wt% and C elemental composition

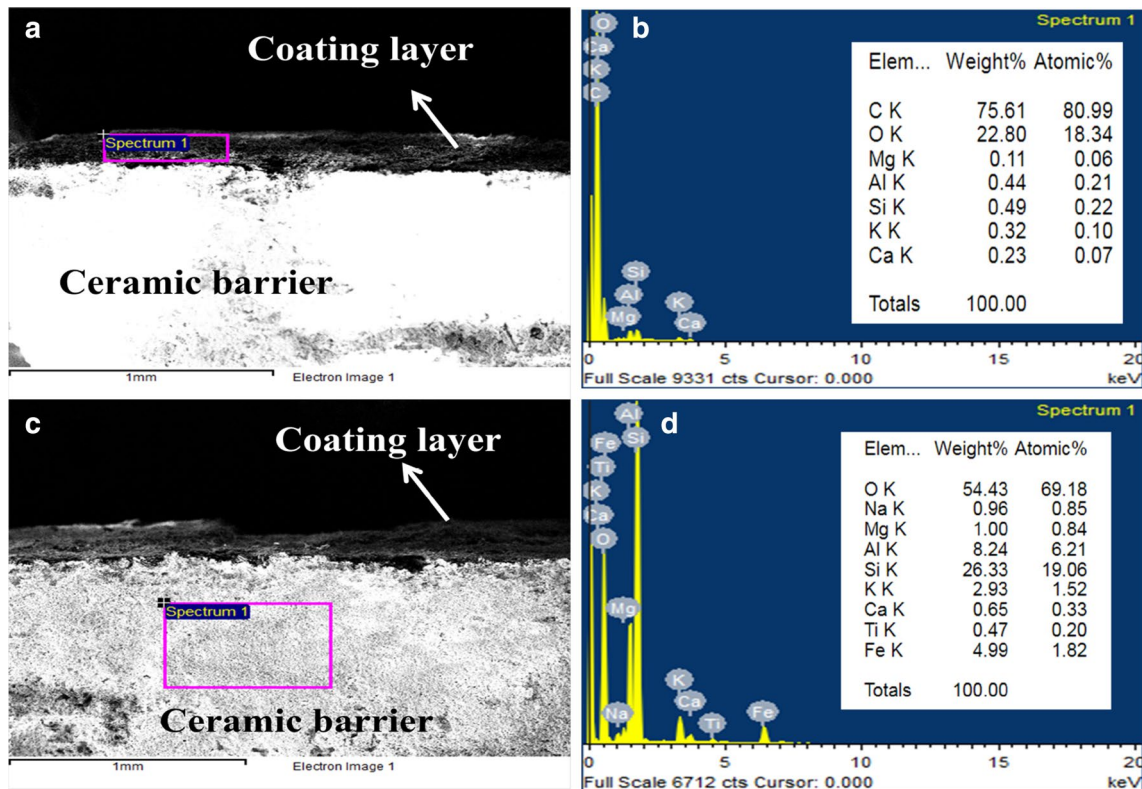


Fig. 5 **a, b** EDS of coating portion and **c, d** EDS of uncoated portion of the RC coated ceramic barrier

is found to be almost nil in the EDS of the other part of the RC coated barrier (Fig. 5c, d). This confirms that the upper layer (coated layer) is mainly composed of carbon.

The advantage of the vacuum-assisted method used in the present work over other coating methods like electrophoretic deposition (EPD), chemical vapour deposition (CVD), spin and dip coating methods is that it is more convenient to utilize for the preparation of larger area films over membranes substrates and its easy operability [9].

The surface of the NAOC coated barrier is shown in Fig. 6. A smooth and uniform distributed layer of NAOC is obtained over the ceramic barrier after deposition of NAOC particles by vacuum assisted deposition method. Aluminum oxyhydroxide nano particles are clearly seen on the top view of the surface (Fig. 6a, b). Side views of the NAOC coated barrier at different resolution showed that a smooth, dense and eventually distributed layer of thickness 44.9 μm deposited on the ceramic surface (Fig. 6c, d). As it can be seen that the roughness of the ceramic barrier (Fig. 4a, b) disappeared on coating of NAOC and a smooth layer is obtained which also showed the effectiveness of the coating.

Two distinct portions in the side view of the coated barrier was analysed by EDS (Fig. 6g, h) and observed that in the topmost portion, elements Al, O and C are found in higher ratios as compared to the Si confirming as the NAOC layer. In the other portion, Si elemental composition is higher as compared to C confirming as the ceramic substrate. This confirms the well deposition of the NAOC particles over the ceramic substrate.

Pure water permeability study is another very important parameter to characterize the barrier quality. Pure water permeability of the ceramic substrate, 0.1 g RC and NAOC coated ceramic substrate was determined by allowing deionized water to pass through the barrier at different applied pressure. It is observed that the pure water flux of the ceramic substrate and its coated from is directly proportional to the pressure drop across the barriers in accordance with Darcy's law (Eq. 1).

$$J_W = L_p \Delta P \quad (1)$$

where J_W = flux, L_p = permeability and ΔP = pressure drop.

From Fig. 7, it is seen that in case of the RC coated ceramic substrate permeability decreases to almost half

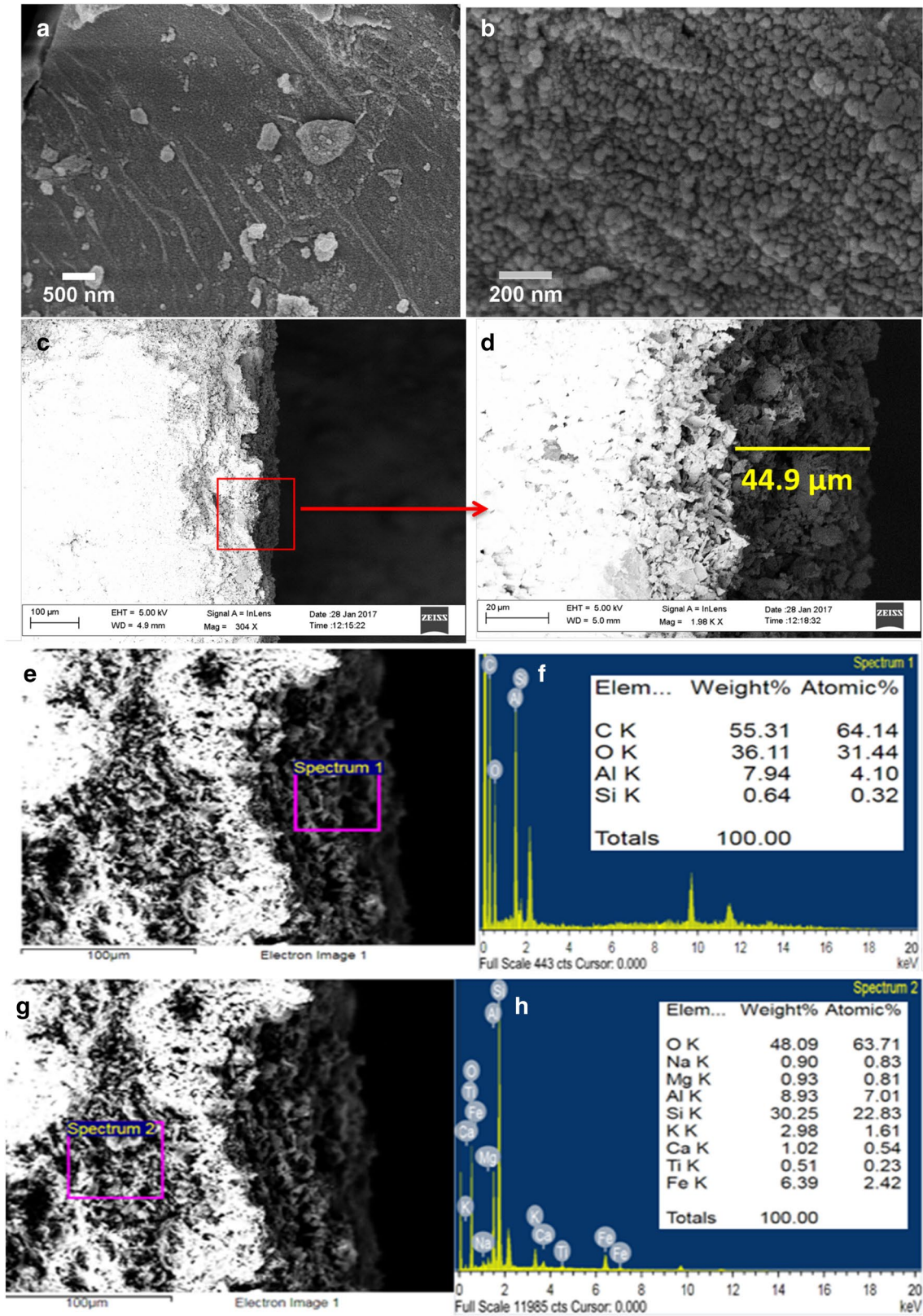


Fig. 6 FESEM images of **a–c** top view and **e, f** cross section of NAOC coated barrier, **d** EDS analysis of top view and **g, h** EDS analysis of cross section of NAOC coated barrier

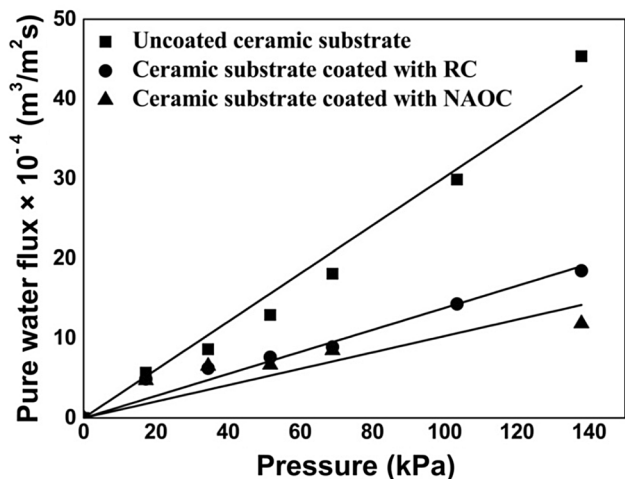


Fig. 7 Comparative permeates flux measurement of ceramic barrier before and after coating

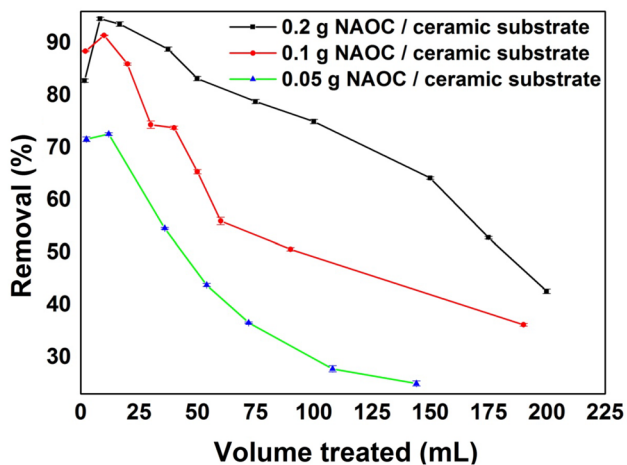


Fig. 8 Competitive breakthrough curves of fluoride removal as a function of coating amount

of the raw ceramic barrier and it further decreased on coating with NAOC.

3.3 Fluoride removal study by coated ceramic barrier

The suitability of the NAOC coated ceramic barrier for defluoridation was studied and the results are presented in Fig. 8. The ability of the coated barrier for fluoride separation was studied by varying the amount of coating and found that defluoridation capacity increased on increasing the amount of coating from 0.05 to 0.2 g per barrier. The flow of the solution through the coated barrier decreases from 1.2 to 0.833 mL min⁻¹ upon increasing amount of coating from 0.05 to 0.2 g per barrier. A plot of percentage

removal versus treated volume with the function of coating amount is shown in Fig. 8. In case of 0.2 g NAOC coated barrier, a constant removal capacity of more than 83% is obtained from 5 mg L⁻¹ synthetic fluoride solution and decreases slowly after treatment of 50 mL fluoride solution. In case of the 0.05 g coated barrier, defluoridation capacity gradually decreases to 25% from initial 71% in 120 min. From Table S.4, on comparing the defluoridation capacity of different types of barriers used in the study is compared after the treatment of 50 mL fluoride contaminated solutions. It is observed that the uncoated and the RC coated barriers are not able to remove fluoride from the synthetic 5 mg L⁻¹ fluoride solution where as on coating with 0.05 g NAOC, the defluoridation capacity of the barrier increased to 46.06% and further increases to 83.15% on coating with 0.2 g NAOC.

The possible fluoride adsorption mechanism in the NAOC coated barrier can be explained as a result of electrostatic interaction between AlOOH and fluoride ions caused by the protonation of hydroxyl group of AlOOH (Eq. 2) followed by ligand exchange reaction (Eqs. 3-4) [1, 19, 25]. Aluminium-fluoride (=Al ··· F) electrostatic interaction is a strong interaction due to hard acid-hard base type of interactions which is responsible for anchoring fluoride ions in the aluminium centres.

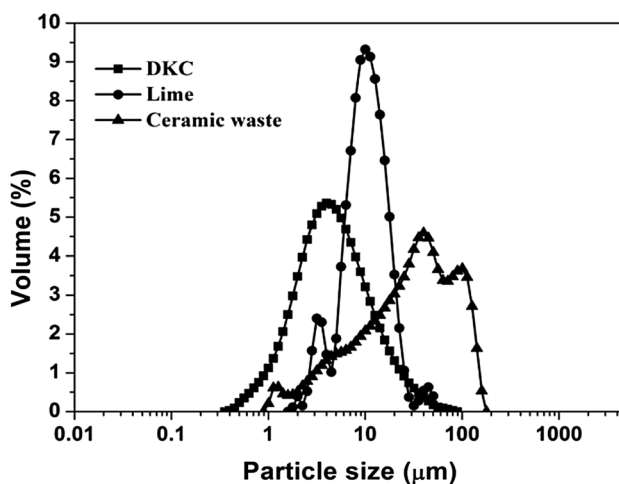
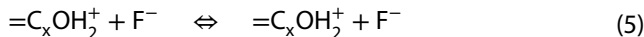
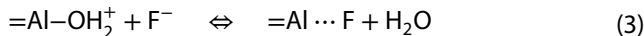
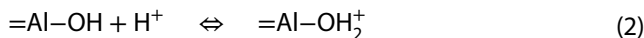


Fig. 9 Particle size analysis of lime, clay and spent adsorbent used for clinker preparation

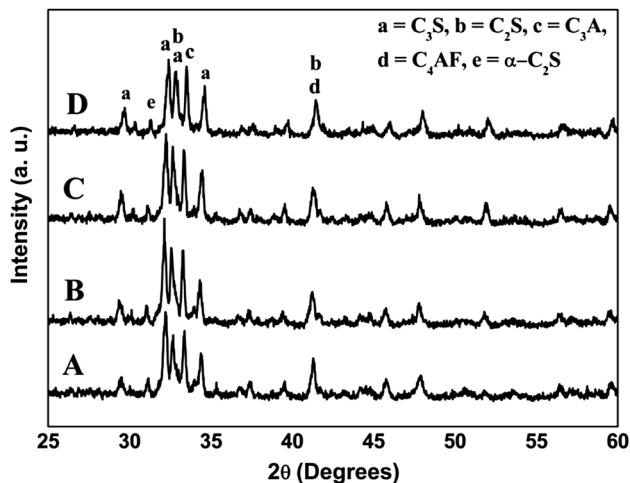


Fig. 10 PXRD pattern of Clinker prepared from lime, clay and spent adsorbent, **a** CM-1450, **b** FCM-1450, **c** CM-1350 and **d** FCM-1350

The adsorption process is believed to enhance by the adsorption of fluoride ions on the oxygen containing functional groups present in the carbon material (Eq. 5).

3.4 Permanent immobilization of ceramic waste

The waste generated after successful separation of toxic fluoride were used to partially substitute the clay portion of the raw mixture of clinker prepared by black meal method. By this method we tried to use the fluoride adsorbed spent ceramic barriers to prepare cement clinkers keeping in mind the value addition as well as permanently immobilize the toxic fluoride in cement clinkers. The particle size of the raw materials used for clinker preparation is presented in Fig. 9. For all the three samples, particle size ranged from 0.3 to 200 μm .

The necessary phases observed in clinkers fired in 1450 $^{\circ}\text{C}$ and 1350 $^{\circ}\text{C}$ where phases like tricalcium silicate, C_3S ($3\text{CaO}\cdot\text{SiO}_2$), di calcium silicate, C_2S ($2\text{CaO}\cdot\text{SiO}_2$), tri calcium aluminate, C_3A ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) and tetra calcium aluminoferrite, C_4AF ($4\text{CaO}\cdot\text{Al}_2\text{O}_3\text{Fe}_2\text{O}_3$), were found in the XRD patterns (Fig. 10). On lowering the burning temperature by 100 $^{\circ}\text{C}$ not much difference in the peaks were observed other than slightly decrease in the intensities. The decrease in the burning temperature from the conventional 1450 $^{\circ}\text{C}$ by 100 $^{\circ}\text{C}$ is due to the addition of the ceramic waste; aluminium and fluoride are reported as mineralisers for cement production [14]. In our case the spent adsorbent contains fluoride and high amount of aluminium which helps in reducing the burning temperature of the cement raw nodules to obtain good quality clinkers.

From fluoride leaching experiment it was observed that the fluoride concentration in the leachate solution is nil which suggests that leaching of fluoride from the prepared

clinker materials is nil or it is in very small quantity which is below the detected limit of the ion sensitive electrode (Orion 4 star, pH/ISE). Lower fluoride content in the leachate suggests that that the ceramic fluoride waste can be used for partial replacement of clay in cement clinker preparation.

4 Conclusions

In this work, an easy and efficient method is applied successfully for coating of ceramic substrate by vacuum assisted slurry deposition method. The coated ceramic barriers are characterized well by FESEM and EDS technique and found that a smooth and effective coating of carbon particles can be obtained in this method. The NAOC coated barriers were used for fluoride separation from contaminated water with very good efficiency. It is also shown that the ceramic waste obtained after successful adsorption of the fluoride ions can be used in cement clinker preparation by black meal method in which clay composition of the raw mixture can be partially substituted by spent ceramic waste. It was also observed that addition of fluoride adsorbed barriers can reduce the conventional firing temperature of the clinkers because of its mineralizing property. Necessary phases required for cement clinker formation was obtained and leaching studies showed that the method could be a promising way to permanently immobilize the fluoride bearing ceramic and clay wastes with an advantage of value addition.

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Compliance with ethical standards

Conflict of interest The authors declare no competing financial and academic interest.

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