Journal of Hazardous Materials 192 (2011) 108–115

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Processed wastewater sludge for improvement of mechanical properties of concretes

Carlos Barrera-Díaz [a,*], Gonzalo Martínez-Barrera [b], Osman Gencel [c], Lina A. Bernal-Martínez [a], Witold Brostow [d]

[a] Centro Conjunto de Investigación en Química Sustentable, Universidad Autónoma del Estado de México – Universidad Nacional Autónoma de México (UAEM-UNAM), Carretera Toluca-Atlacomulco, km 14.5, Unidad El Rosedal, C.P. 50200, Toluca, Edo. de México, Mexico
[b] Laboratorio de Investigación y Desarrollo de Materiales Avanzados (LIDMA), Facultad de Química, Universidad Autónoma del Estado de México, Carretera Toluca-Atlacomulco, Km.12, San Cayetano C.P. 50200, Toluca, Edo. de México, Mexico
[c] Civil Engineering Department, Faculty of Engineering, Bartin University, 74100 Bartin, Turkey
[d] Laboratory of Advanced Polymers & Optimized Materials (LAPOM), Department of Materials Science and Engineering and Center for Advanced Research and Technology (CART), University of North Texas, 1150 Union Circle # 305310, Denton, TX 76203-5017, USA

ARTICLE INFO

Article history:
Received 11 January 2011
Received in revised form 25 April 2011
Accepted 28 April 2011
Available online 5 May 2011

Keywords:
Concrete
Construction materials
Compression properties
Electrocoagulation
Wastewater treatment

ABSTRACT

Two problems are addressed simultaneously. One is the utilisation of sludge from the treatment of wastewater. The other is the modification of the mechanical properties of concrete. The sludge was subjected to two series of treatments. In one series, coagulants were used, including ferrous sulphate, aluminium sulphate or aluminium polyhydroxychloride. In the other series, an electrochemical treatment was applied with several starting values of pH. Then, concretes consisting of a cement matrix, silica sand, marble and one of the sludges were developed. Specimens without sludge were prepared for comparison. Curing times and aggregate concentrations were varied. The compressive strength, compressive strain at yield point, and static and dynamic elastic moduli were determined. Diagrams of the compressive strength and compressive strain at the yield point as a function of time passed through the minima as a function of time for concretes containing sludge; therefore, the presence of sludge has beneficial effects on the long term properties. Some morphological changes caused by the presence of sludge are seen in scanning electron microscopy. A way of utilising sludge is thus provided together with a way to improve the compressive strain at yield point of concrete.

1. Introduction

With the current emphasis on environmental health and water pollution issues, there is an increasing awareness of the need to dispose of wastewater safely and beneficially. Properly planned use of sewage wastewater and its byproducts alleviates a number of environmental problems [1]. Approximately 99% of the wastewater stream that enters a wastewater treatment plant is discharged as treated water. The remainder is a dilute suspension of solids captured by the treatment process-commonly referred to as sludge [2].

Sludge is an unavoidable byproduct of primary, secondary, and advanced wastewater treatment processes. It is typically generated at a rate of 70–90 g/person equivalent per day [3–5]. Sludge contains between 1 and 4 wt% of solid materials, and the rest is water. Importantly, sludge contains pollutants and unstable pathogen content, therefore leading to potential health and environmental hazards [6,7]. The most widely used final disposal method of sludge is putting it into landfills, with all the risk of soil contamination and degradation of the urban landscape [8].

There are many sludge treatments, and the most common ones are stabilisation and dewatering. Sludge stabilisation can be performed by thermal, chemical or biological treatment. Dewatering of sludge can be achieved by mechanical and thermal means. Mechanical dewatering consists of processes like gravitation settling, centrifugation and filtration (belt, vacuum or pressure). However, those treatments and the related sludge transport are costly [9–14]. Moreover, another treatment involves an incineration process which is possible to obtain incinerator sewage sludge ash with pozzolanic properties; such waste contains significant levels of phosphates. Acid washed residue is produced when the incinerator sludge ash is submitted to acid washing; such residue has potential to be used in construction products [15,16].

Compositions of sludge vary considerably depending on the wastewater composition and the treatment processes used [17]; electrochemical technologies produce up to 50% less sludge than

* Corresponding author. Tel.: +52 722 2173890; fax: +52 722 2175109.
E-mail addresses: cbd0044@yahoo.com, cbarrera@uaemex.mx (C. Barrera-Díaz).

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doi:10.1016/j.jhazmat.2011.04.103

conventional treatments. Electrocoagulation technology removes metals, colloidal solids and particles, and soluble inorganic pollutants from aqueous media by introducing highly charged metal hydroxide species. These species neutralise the electrostatic charges on suspended solid particles, facilitating agglomeration or coagulation and subsequent separation from the aqueous phase. The treatment prompts the precipitation of certain metals and salts; the amount of sludge produced is smaller when compared with ordinary coagulation. For example, the sludge formed in the electrocoagulation method with iron contains a higher content of dry and hydrophobic solids than that produced in coagulation by the action of $FeCl_3$ followed by the addition of NaOH or lime. The advantages are that operating costs are much lower than in most conventional technologies [18,19].

Cements and concretes are manufactured in very large quantities for construction and other industries [20–24]. The idea of using sludge as a component of cement has already been explored to some extent, notably by two Japanese groups [25,26]. The pulp and paper industry has reported making building blocks and panels using sludge [27,28]. Moreover, sludge can be combined with cellulose fibres in the cement matrix to create so-called hempcrete, with high compression strength, good impact resistance, cohesion and workability. The use of sludge reduces costs because of the lower consumption of water, clay and electricity in the production process [29].

The elastic moduli is the most often used characteristics of composites. In the case of building structures, the non-destructive tests (NDTs) take into account the acoustic impedance of the system components – important factors influencing ultrasonic wave propagation [30,31]. In general, dynamic elastic moduli (E_d) serve well for hard as well as fragile materials. E_d is determined by measuring the pulse velocity along the composite and using electrical transducers located on the opposite sides of the cylindrical specimens of concrete. The energy supplied the ultrasound depends on how compact the composite is, including the content of binder and filler as well as content of pores. E_d can be calculated as:

$$E_d = u^2 w \frac{(1+\nu)(1-2\nu)}{(1-\nu)} \quad (1)$$

Here u is the pulse velocity; w is the weight of the concrete specimen; and ν is the Poisson ratio. E_d depends on the component properties of the aggregates and their relationship with the cement.

Therefore, wastewater sludge was subjected to a variety of treatments – chemical and electrochemical – and the effects of the inclusion of sludge on the properties of concretes were investigated.

2. Materials and methods

2.1. Wastewater samples

Samples of industrial wastewater were collected in a biological reactor from a treatment plant located at the end of an industrial park; the park receives discharges from 144 different facilities. The samples were collected in plastic containers and cooled to 4 °C. Parameters characterising the wastewater prior to treatment (raw water) were a chemical oxygen demand (COD) of 410 mg L^{-1}, a colour of 520 platinum–cobalt units (Pt–Co), a turbidity of 55 nephelometric turbidity units (NTU) and pH = 7.5.

2.2. Sludge from chemical treatment

Several processes are used for the separation of solid suspensions [32]. Such separation can also be achieved without external agents under the effect of gravitational forces alone, but it takes much more time. Widely used processes include coagulation [33,34] and flocculation [35–37]. Coagulation is based on neutralisation of the electrostatic repulsion between particles with the same electric charge by the introduction of particles with the opposite charge. The efficacy of coagulants used is evaluated by performing a jar test in which the turbidity is measured as a function of polymer concentration. A TEMSA-JTR 1010 apparatus from TEMSA, S.A. de C.V., Mexico was used, comprising six paddle rotors equipped with six beakers of 1.0 L each so that 1 L of the sample is put in each beaker. Three coagulants were applied: ferrous sulphate ($FeSO_4$), aluminium sulphate ($Al_2(SO_4)_3$) and aluminium polyhydroxychloride (PAC). The respective compositions are listed in Tables 1 and 2. The procedure was as follows: a rotation speed of 115 rpm for 1 min, then 30 rpm for 10 min and sedimentation for 15 min. The final gravity settling stage lasted for another 1 h before sampling for COD analysis. The tests were carried out at a constant pH = 7.5 ± 0.5.

Table 1
Percentage of COD removal (mg L^{-1}) as a function of coagulants.

Coagulant dosage (mL L^{-1})	$FeSO_4$ 0.1 M	$Al_2(SO_4)_3$ 0.1 M	PAC
0	–	–	–
0.5	–	–	2.4
1.0	–	–	12.2
1.5	–	–	20.3
2.0	–	–	29.0
2.5	3.7	5.6	41.0
5	7.8	12.2	–
7.5	14.9	17.1	–
10	25.4	31.5	–
12.5	31.0	37.1	–

2.3. Sludge from electrochemical treatment

The treatment was performed in a batch electrochemical reactor. The reactor cell contains an array of 6 parallel monopolar iron electrodes; each electrode is 5.5 cm × 3.0 cm, and the surface area is 16.5 cm^2. The total anodic surface A_a was 99 cm^2. The capacity of the reactor was 1.5 L, and a direct-current power source supplied the system with 1–4 A, corresponding to current densities of 10–40 mA cm^{-2} [32]. It is well known that the initial pH of the sample can have either a positive or negative influence on the electrochemical treatment results. Hence, after the samples were introduced into the reactor, the pH was initially adjusted using either NaOH or H_2SO_4 before the electrical current was applied. In these experiments, pH values of 3, 5, 7 and 9 were applied. Then, the sludge samples were air-dried at room temperature (18.0 ± 0.5 °C) and collected for subsequent application.

2.4. Concrete specimen preparation

For preparing the concrete specimens, sand as natural silica, marble from a local company (GOSA^{TM}, Atizapan, Mexico) and Portland cement (Cruz Azul^{TM}, Monterrey, Mexico) Type II were used.

Table 2
COD (mg L^{-1}) as a function of coagulants.

Coagulant dosage (mL L^{-1})	$FeSO_4$ 0.1 M	$Al_2(SO_4)_3$ 0.1 M	PAC
0	410	410	410
0.5	–	–	400
1.0	–	–	360
1.5	–	–	327
2.0	–	–	291
2.5	395	387	242
5	378	360	–
7.5	349	340	–
10	306	281	–
12.5	283	258	–

Table 3
Mixture proportions of the concrete ($kg\,m^{-3}$).

Codes	Cement	Water	Sludge	Silica sand	Marble
CA-T	450	337.5	–	675	1125
CA-S	450	–	337.5	675	1125
CB-T	450	337.5	–	900	1350
CB-S	450	–	337.5	900	1350

The Portland cement and the silica sand were sieved through a 100 mesh (150 μm), and, for marble, the 14 mesh (1.4 mm) was used. The specimens were prepared separately on different days, in cylindrical moulds (2″ diameters and 4″ long) and placed in a controlled temperature room at 23.0 ± 3.0 °C with the surface exposed to moisture in air and no less than 50% humidity according to the ASTM C-511 standard. The curing times were 7, 14, 21 or 28 days, according to the ATSM C31 standard [38,39].

For each cubic meter of concrete, concrete composition was given in Table 3. Binder content and water-binder ratio was 450 $kg\,m^{-3}$ and 0.75, respectively. Sludge was replaced with water. Four different series of concrete specimens were prepared, called CA-T, CA-S, CB-T and CB-S, as shown in Table 3. CA and CB means "Concentration A" and "Concentration B", respectively, and T means "Target" is to say without sludge, in contrast to S referred to "Sludge"; Four different lots were made on different days, each containing five samples.

2.5. Mechanical testing of concretes

Compressive tests of concrete cylindrical specimens were carried out in a universal testing machine Model 70-S17C2 (Controls™, Cernusco, Italy) according to the ASTM C-39 standard. The dynamic elastic modulae E_d of concrete cylindrical specimens were determined on the basis of ultrasound propagation using equipment for construction materials: an Ultrasonic Pulse Velocity Tester model 58-E0048 (Controls™, Cernusco, Italy) with an ultrasonic resolution of 0.1 ms.

2.6. Morphological characterisation

The sludge generated by the electrochemical process and the fractured concrete specimens were analysed by scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDS). A JEOL JSM-6510LV microscope was used. EDS was performed with INCA pentaFETX3, Oxford, UK, machine to provide elemental analysis in situ.

3. Results and discussion

3.1. Chemical treatment of sludge

The COD and COD removal percentage results are listed in Tables 1 and 2, respectively, in which coagulant was used up to dosages of 12.5 $mL\,L^{-1}$. The COD value goes down from 410 to 283 $mg\,L^{-1}$ when $FeSO_4$ is used; which means a reduction of 31% of COD. When $Al_2(SO_4)_3$ is used, the COD decreases from 410 to 258 $mg\,L^{-1}$, which is a reduction of 37%. Finally, when PAC is used, the reduction of COD is from 410 to 242 $mg\,L^{-1}$. This last case shows a reduction by 41% while the concentration of such coagulant amounts to only 20% of the concentration of the earlier two coagulants.

PAC is a pre-polymerised Al (III) chemical containing a range of hydrolysed and polymeric species that are relatively large and carry high cationic charges. Their enhanced surface activity and improved charge neutralising capacity make them effective at low doses. Therefore, PAC has several advantages over conventional Al (III) salts: rapid aggregation velocity, formation of larger and heavier solid suspension particles and lower required dosage. Therefore, PAC has been used extensively in wastewater treatment plants. Chemical coagulation introduces a large quantity of iron into the solution, which will be present in the residual sludge.

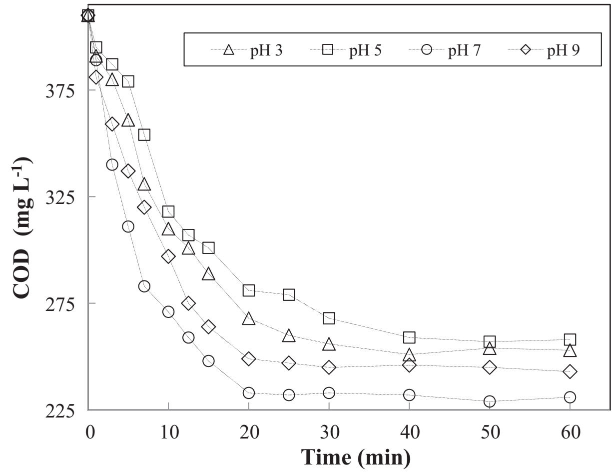

Fig. 1. Removal of COD ($mg\,L^{-1}$), from wastewater at pH = in turn 3, 5, 7 and 9 as a function of electrochemical treatment time when obtaining the sludge at 40 $mA\,cm^{-2}$.

3.2. Electrochemical treatment of sludge

As mentioned in Section 2.3, pH values of 3, 5, 7 and 9 were applied. Fig. 1 shows the COD as a function of treatment time at different initial pH values. After 30 min or so, the maximum COD reduction occurs at pH = 7 (43%), followed by that at pH = 9 (40%) and then at pH = 3 and 5 (39% or 37%). To explain these results, consider a treatment used to dissolve artificial anodes of iron or steel immersed in polluted water, giving rise to the corresponding metal ions that yield different Fe(II) and/or Fe(III) species depending on the pH of the medium [40]. Mechanisms for the removal of pollutants by electrocoagulation will be explained with two specific examples involving iron because this metal has been extensively used to clean wastewater [19].Anode:

$$Fe_{(s)} \rightarrow Fe_{(aq)}{}^{2+} + 2e^- \quad (2)$$

$$Fe_{(aq)}{}^{2+} + 2OH_{(aq)}{}^{-} \rightarrow Fe(OH)_{2(s)} \quad (3)$$

Cathode:

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2(g)}2OH_{(aq)}{}^{-} \quad (4)$$

Overall:

$$Fe_{(s)} + 2H_2O_{(l)} \rightarrow Fe(OH)_{2(s)} + H_{2(g)} \quad (5)$$

Eq. (2) is favoured at pH < 5.0 because of the chemical attack of protons so that predominantly monomeric species like Fe^{2+} can be expected in solution. According to Eq. (3), an increase in pH is expected to lead to the formation of insoluble iron hydroxide. The hydroxide should be able to remove contaminants by surface complexation [41] followed by coagulation [42,43]. Namely, the pollutant acts as a ligand (L) to chemically bind hydrous iron:

$$L{-}H_{(aq)}(OH)OFe_{(s)} \rightarrow L{-}OFe_{(s)} + H_2O_l \quad (6)$$

Prehydrolysis of Fe^{3+} cations also leads to the formation of reactive clusters for water treatment. Structural models for these oxyhydroxy iron cations have been extensively reported in the literature [34]. In the electrocoagulation treatment with iron, various species are formed such as $Fe(OH)_4{}^{-}$, $Fe(H_2O)_3(OH)_3$,

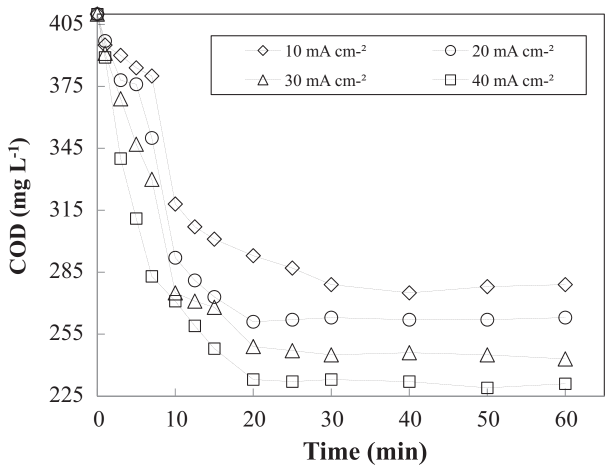

Fig. 2. Reduction of COD as a function of electrochemical treatment time at current densities of 10, 20, 30, and 40 mA cm^{-2} at pH = 7 while obtaining the sludge.

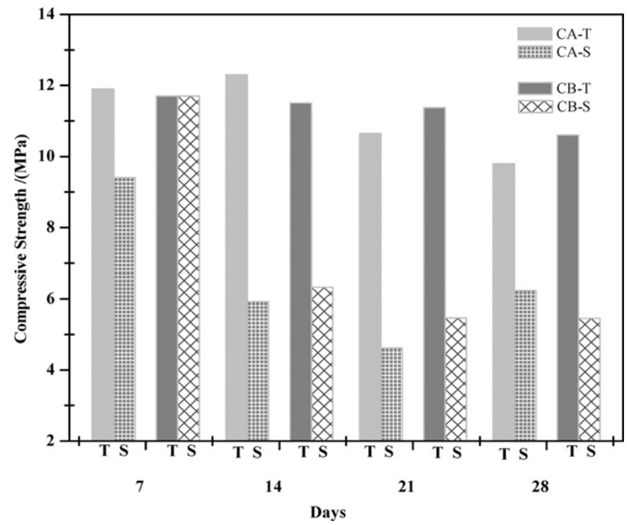

Fig. 3. Compressive strength of the concrete at different curing days.

$Fe(H_2O)_6{}^{3+}$, $Fe(H_2O)_5(OH)^{2+}$, $Fe(H_2O)_4(OH)^{2+}$, $Fe_2(H_2O)_8(OH)_2{}^{4+}$, and $Fe(H_2O)_6(OH)_4{}^{4+}$ [39].

Fig. 2 shows the COD as a function of treatment time for different applied current densities at pH = 7. The applied current density was varied from 10 to 40 mA cm^{-2}. The results can be explained as follows. An increase in the current density leads to an increase in the amount of oxidised irons generated from the electrode; see Eq. (6). As expected, higher current densities cause larger reductions in COD; at 10 mA cm^{-2}, the reduction amounts to 33%; at 20 mA cm^{-2}, it is 36%; at 30 mA cm^{-2}, it is 40%; and, at 40 mA cm^{-2}, it is 43%. However, as the current density increased, the applied potential increased as well. It is advisable to limit the current density to avoid adverse effects such as heat generation. From Eqs. (2)–(5), one can infer that the sludge production increases with increasing current density; for 10 mA cm^{-2}, it is 4 kg m^{-3}; for 20 mA cm^{-2}, it is 5 kg m^{-3}; and, for 30 mA cm^{-2}, it is 8 Kg m^{-3}. These results agree with literature values [44].

After the electrochemical treatment, the effluent was left to settle for 30 min, and the water was filtered to obtain the settleable solids. The amount of dry sludge obtained from electrochemical treatment was ≈10 kg m^{-3}. Biological systems produce from 45 to 65 kg m^{-3-} while chemicals treatments range from 15 to 25 kg m^{-3}. Our electrochemically produced sludge is more compact and takes less space than sludge obtained using conventional or chemical treatments. In other respects, this sludge is comparable to those produced by other treatments [32,45–48].

Sludge formed via the electrocoagulation method with Fe contains a higher content of dry and hydrophobic solids than those produced by coagulation by the action of FeCl followed by the addition of NaOH or lime. Traditional coagulation generates sludge that is not compact and requires large volumes. Management and final disposal of such residues is difficult and rather expensive [49,50].

3.3. Mechanical properties of concretes

3.3.1. Compressive strength

The compressive strength values of the concretes are presented in Fig. 3 as a block diagram for 7, 14, 21 and 28 days (the standard period of 28 days is covered). The values for concrete without sludge (CA-T and CB-T) varying from 9.7 to 12.3 MPa, while concrete with sludge (CA-S and CB-S) from 4.6 to 9.4 MPa; this means a maximum reduction of 110% when the sludge is added.

The concretes without sludge show moderate changes with time; the values for both CA-T and CB-T are similar. Moreover, when adding more marble and sand concentration (as CB-T), the diminution on compressive strength is less. The behaviour of concretes with sludge is different. The compressive strength of the CA-S sample passes through a minimum at 21 days and after increase for 28 days. But the CB-S samples diminish progressively their values with time. Such behaviour can be related to the presence of non-degradable organic matter in the sludge after electrochemical treatment, which in principle can reduce the cement hydration; microcalorimetric measurements show that the cement hydration can take years [51].

The strength of materials can be improved by blending, insertion of fibres, gamma irradiation, using fillers and/or combinations of these approached [52–76]. Smaller particles usually provide more reinforcement. Therefore, while the CA samples contain less marble than the CB samples, apparently, marble particles in CA are better dispersed.

3.3.2. Compressive strain at the yield point

The results for the compressive strain at the yield point are presented in Fig. 4. The values for concretes without sludge vary from 0.009 to 0.012 mm mm^{-1}. For concretes with sludge, the values are lower, from 0.007 to 0.010 mm mm^{-1}. Both ranges of values are higher than standard values for Portland cement concrete (0.003 mm mm^{-1}).

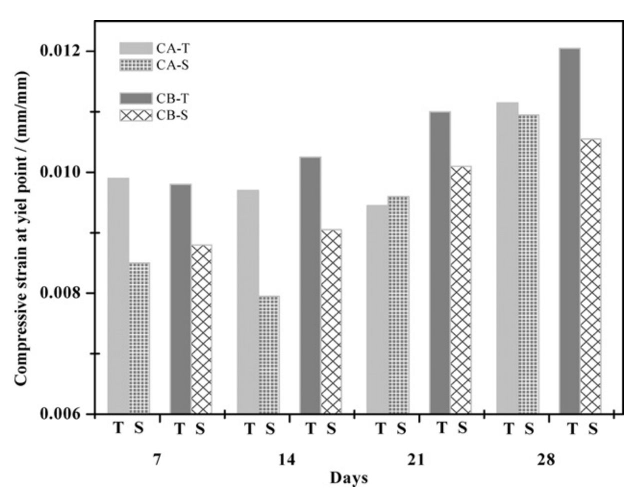

Fig. 4. Compressive strain at yield point of concrete at different curing days.

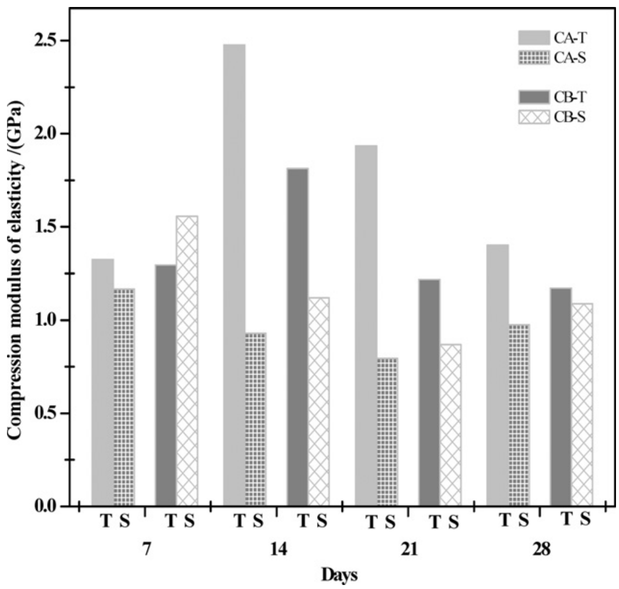

Fig. 5. Compression modulus of elasticity of the concrete at different curing days.

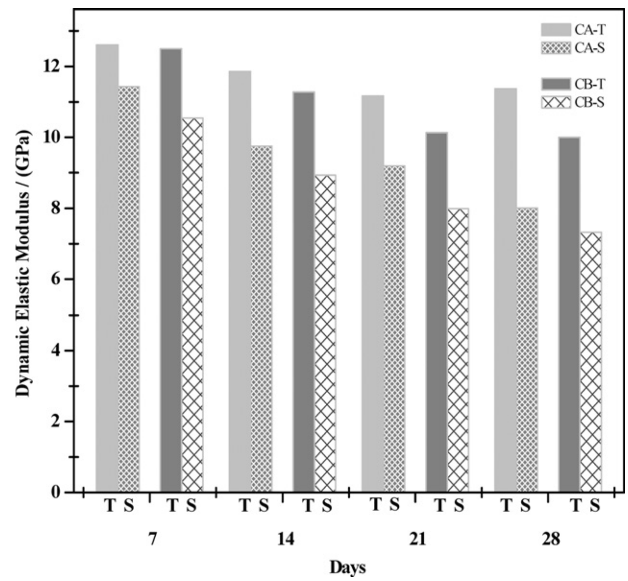

Fig. 6. Dynamic elastic modulus of concrete at different curing days.

Two different types of behaviour for concretes without sludge are seen. For CA-T samples, a minimum at 14 days is required. For CB-T samples, there is a continuous increase of the compressive strain as a function of time. In terms of time dependence, there is no difference between specimens with and without sludge. In spite of passing through a minimum, the strain after 28 days for the CA-S sample is higher than for the CB-S sample.

It is interesting to compare these results for mineral concretes with those for polymer concretes. The latter are particulate composites in which the polymeric resin (for example, an unsaturated polyester) binds inorganic aggregates (for example, silica sand or marble) instead of water and cement binder typically used in Portland cement concretes. Therefore, for polymer concretes with marble particles, the compressive strain at yield varies from 0.007 to 0.020 mm mm^{-1} [74]. For polymer concretes with silica sand as aggregate, the respective values are in the range from 0.010 to 0.014 mm mm^{-1} [64]. Therefore, this sludge, which constitutes a minority component, plays a similar role in enhancing the compressive strain values with respect to ordinary Portland cement concrete, as does the polymeric matrix in polymer concretes.

3.3.3. Compression moduli of elasticity

Fig. 5 shows the compression moduli of elasticity E_c of concretes as a function of time. For concretes without sludge the values varying from 1.21 to 2.47 GPa, while for concretes with sludge from 0.79 to 1.55 GPa, this means a maximum diminution of 53% when sludge is added. The time dependence of concretes without sludge (CA-T and CB-T) shows a maximum at 14 days and after decreasing with time. In contrast for concretes with sludge (CA-S and CB-S) a minimum value is obtained at 21 days and after increasing for 28 days. Therefore, a further increase of E_c in concretes with sludge is expected, in consequence higher elasticity values and a more hard material.

3.3.4. Dynamic moduli of elasticity

These values are based on ultrasound velocity measurements. The behaviour of the dynamic elastic moduli E_d is different than that of compression moduli; compare Figs. 5 and 6. All specimens exhibit the same behaviour of E_d: the values decrease with time. After 28 days, the highest value is for concrete without sludge (CA-T), namely 11.6 GPa. The values for concrete without sludge (CA-T and CB-T) varying from 10.0 to 12.6 GPa, while for concrete with sludge (CA-S and CB-S) from 7.3 to 11.4 GPa, this means a maximum diminution of 36% when sludge is added.

In general, higher values for CB are found when comparing with CA type, independently of add or not sludge. We argue that higher quantities of aggregates (marble and sand) provoke lower elastic

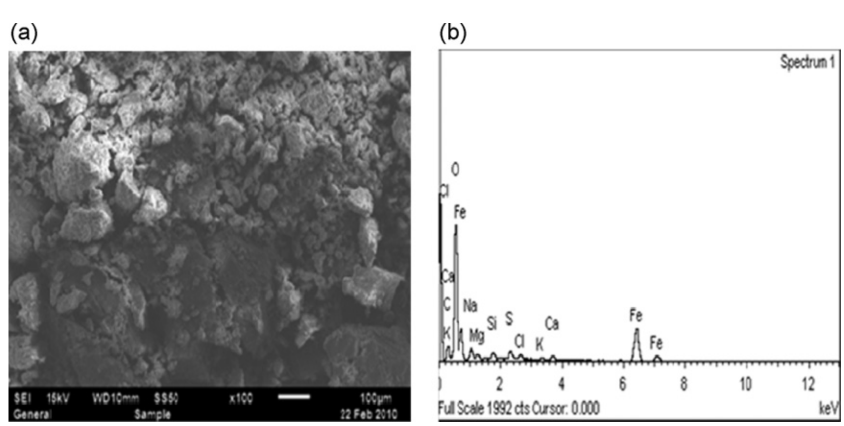

Fig. 7. (a) SEM image of the morphology of dry sludge (100×), and (b) microanalysis image (EDS) of dry sludge.

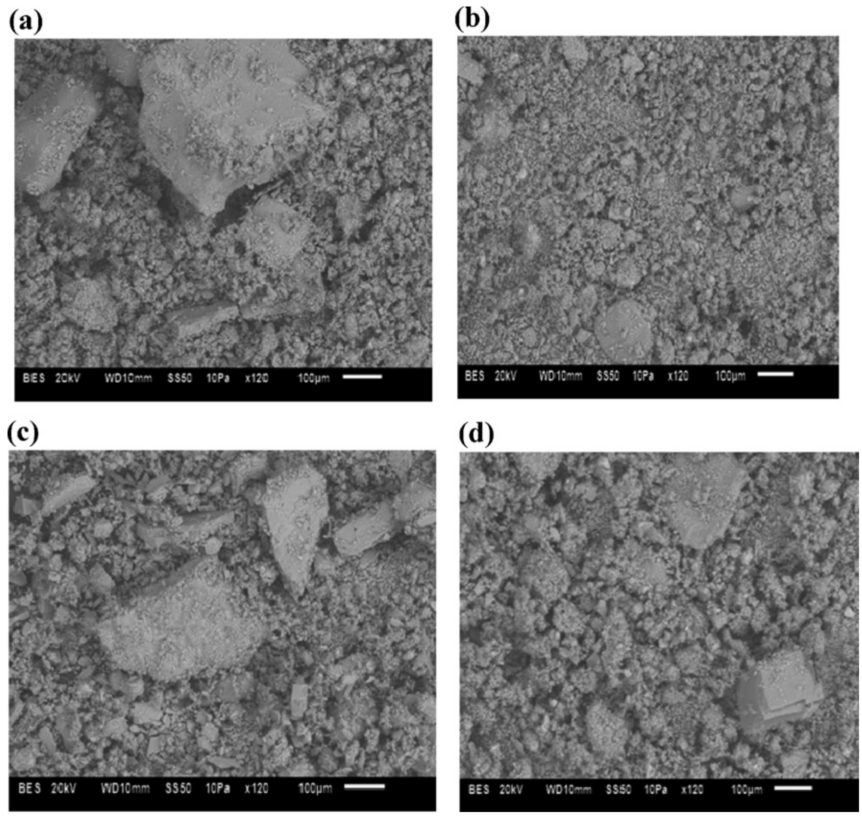

Fig. 8. SEM image of the curing concrete at 7 days after compressive testing: (a) CA-T, (b) CA-S, (c) CB-T and (d) CB-S.

moduli and in consequence a more ductile material is obtained [38,39].

3.4. Morphological characterisation

The SEM and EDS results for a sludge generated by the electrochemical process at pH = 7 are reported here. The sludge shows a heterogeneous morphology, as shown in Fig. 7a. Its elemental composition as determined by EDS includes carbon (20.9%), oxygen (46.9%), iron (16.2%), and sodium (2.3%) as the main elements (see Fig. 7b and Table 4).

3.5. Fracture surfaces of concretes

Fig. 8 shows the morphology of the fractured surfaces of the concretes subjected after seven days to compressive testing. For the CA samples, the surface of the sludge containing material (Fig. 8b) is less rough than the specimen without sludge (Fig. 8a). The sludge seems to acts as a wrapper for the marble particles. For the CB samples, both surfaces are fairly similar (Fig. 8c and d). Somewhat larger marble particles are seen in the absence of sludge. From Fig. 8d, it is inferred that the amount of sludge is insufficient to cover all silica sand particles.

Table 4
Elemental composition (EDS) the sludge.

Element	Weight %
C	20.95
O	46.90
Na	2.26
Mg	0.61
Si	0.79
P	0.27
S	1.19
Cl	0.59
K	0.38
Ca	0.49
Fe	16.20
Totals	90.63

4. Conclusions

In the electrocoagulation requires simple equipment and is easy to operate with sufficient operational latitude to handle most problems encountered on running. The production of sludge using electrochemical treatment (10 Kg m^{-3}) is viable for reuse, because it is a mud with less organic pollutants compared with other sludges obtained by other physicochemical processes of wastewater treatment. Therefore the sludge production can be used as the component raw materials, for fabrication of civil construction materials. The sludge containing concretes appears economically attractive given the low prices of raw industrial wastes. Wide scale application of this method can significantly improve the environmental situation in industrial regions. The application of sludge can significantly reduce the sludge disposal cost component of sewerage treatment. As expected, mechanical properties depend on the silica sand, marble and sludge concentration. Higher concentrations of silica sand and marble particles create less space in the concrete and in consequence the compressive strength and the compression strain increase; but lower values on the E_c and E_d moduli are obtained. Moreover, when the sludge is added the mechanical features have lower values.

Acknowledgments

The authors wish to acknowledge the support given by the Centro de Investigacion en Quimica Sustentable UAEM-UNAM, Toluca; and also from the Sistema Nacional de Investigadores (SNI), Mexico, DF. Financial support of the Consejo Nacional de Ciencia y Tecnologia (CONACYT), Mexico by Grant # 49899 is acknowledged.

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