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Encapsulating fly ash and acidic process waste water in brick structure

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ABSTRACT

Fly ash contains metals such as cadmium, iron, lead, aluminum and zinc in its structure in appreciable amounts. These metals can leach out into surface and ground waters if fly ash is not properly disposed of. A similar problem also exists for acidic process waste waters discharged by numerous industries. The purpose of this study was to utilize such wastes as additives in the production of construction quality bricks for the purpose of waste elimination. The bricks produced were subjected to flexural strength and water retention capacity tests along with heavy metal leaching experiments in order to determine the applicability of the procedure and the best possible recipes. This paper summarizes the results obtained in these tests along with the possible mechanisms involved in stabilizing the two wastes in the brick structure.

1. Introduction

Fly ash is a pozzolanic material generated from coal-burning thermal power plants. Though a significant fraction of coal fly ash is used as a cement and concrete additive in the world, only a very small portion of the 15 million tons of fly ash generated annually is re-utilized in Turkey [1].

Fly ash contains a range of heavy metals of different mobilities in its structure [2]. Since it is usually disposed of in the form of a slurry in the vicinity of the power plant, fly ash possesses significant environmental risk due to the possibility of leaching of these metals into environment [1,3]. Such risks have paved the way to extensive studies on the physical–chemical properties and leaching behaviour of fly ash [4–10].

There is also a large body of work on the utilization of fly ash in cement and brick production [11–26]. These studies mainly focus on the effect of fly ash addition on the structural behaviour of the final products. In general, it was observed that the properties of the test bricks containing fly ash were found to be at least comparable, if not better than, to those of standard test bricks made without fly ash and met the commercial specifications for fired bricks. Such positive findings suggested the possible use for fly ash as a supplementary material in brick manufacturing, reducing clay consumption. A more recent study [27] focused on the use of fly ash in small amounts (around 5 wt%) to improve the quality of solid bricks. According to this work, the textures of the bricks containing fly ash were very similar to the textures of those without it, except that the samples with the additive contained spherical fly ash particles. They argued that these particles led to a reduction in the density of the bricks and a substantial improvement in their durability, with reduced susceptibility to decay caused by salt crystallization in the pores. This is because fly ash causes a reduction in the number of micropores, the pores that make porous materials most vulnerable to salt-induced decay.

Despite such presence of work on fly ash use in brick manufacturing, there is a need for evaluating the effect of such additives on the stability of the heavy metals contained in the final product structure through systematic leaching studies. Main objective of this study was to investigate the leaching behaviour of heavy metals from bricks which contained two wastes as additives; coal fly ash and acidic process water. The capacity of heavy metals fixation in the structure of the bricks was determined. Mechanical strengths and water retention capacities of the bricks were also measured to determine if there were any detrimental effects of the waste additives on the physical properties of the bricks.

2. Experimental work

2.1. Materials

A clay mixture which was commercially used in a brick factory in Turgulu-Manisa Region of Turkey was utilized as the body material

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Table 1
Chemical composition of the clay and fly ash samples (XRF).

Oxides	%Amount in the sample			
	Fly ash	Clay A (green)	Clay B (red)	Clay C (violet)
SiO_2	55.9	70.5	49.4	60.0
Al_2O_3	16.4	13.3	15.7	13.7
Fe_2O_3	9.9	6.0	6.1	5.9
CaO	6.1	0.5	8.8	5.6
MgO	4.8	1.1	3.8	2.3
K_2O	1.6	1.7	1.7	2.0
Na_2O	0.7	0.5	0.3	0.5
TiO_2	NA	0.8	0.04	NA
MnO	NA	0.6	0.06	NA
Free CaO	0.3	NA	NA	NA
Cl	0.06	NA	NA	NA
SO_3	1.1	NA	NA	0.01

for the bricks. The mixture consists of three different clays to impart desired properties to the final product. The nominal 90% passing size of the clay mixture was 10 μm as determined by Malvern Particle Size Analyzer Master Sizer 2000 HD.

Fly ash used in this study was obtained from the Tuncbilek power station in Western Turkey. The nominal 90% passing size for the fly ash was observed to be 100 μm.

The XRF analysis of the clays and the fly ash is given in Table 1. It can be seen that the ash is rich in CaO which yields strongly alkaline solutions in water (pH higher than 12 in solutions of 1/10 fly ash/water ratio).

The industrial acidic process waste water was collected from a tannery in Izmir, Turkey. The waste water was a black, homogeneous liquid with a total organic content of 2.9 g/L and showed high viscosity. It was acidic with a natural pH of 2.5. Chemical composition determined using ICP-MS (Agilent 7500) after microwave digestion (6 ml HNO_3 and 1 ml of peroxide for 5 ml of waste) is given in Table 2.

Initially, standard bricks were prepared using only the clay mixture for comparison purposes. In the next phase of the study, experimental bricks were prepared by adding predetermined amounts of fly ash and/or acidic process waste water to the clay mixture.

2.2. Methods

2.2.1. Production of bricks

In the production of the standard bricks, a clay mixture called Clay M was prepared by mixing 66% Clay A, 19% Clay B and 15% Clay C. This recipe is commercially used in the brick factory from which these clay samples were obtained. Clay M was mixed with 10% water to produce a green body which was pressed under 15 MPa pressure to produce standard experimental cylindrical bricks of 8 cm diameter 14 cm length and coded as Type S bricks. The experimental green brick bodies containing the fly ash and acidic process waste water were produced using the same conditions but by changing the respective ratios of Clay M, fly ash and acidic process waste water in the overall mixture within a wide range. These bricks were coded as Type A bricks (fly ash amount varied, no acidic process waste water and 10% clean water), Type B bricks (no fly ash, acidic process waste water amount varied and 10% clean water) and Type C bricks (fly ash amount varied, 5% acidic process waste water and 5% clean water). Detailed compositions and coding of the experimental brick samples are listed in Table 3.

Table 2
Chemical composition of acidic process waste water (ICP-MS).

Elements	ppm	Elements	ppb
Na	38.4	Zn	53.7
Al	33.4	Fe	50.6
Mn	9.0	Sr	44.1
Mg	2.0	Li	4.5
Ca	1.5	Cu	2.5
Cr	1.1	Ni	1.8
K	0.8	As	1.2
		Co	0.8
		Cd	0.7

Table 3
Compositions and coding of the brick samples produced.

Brick type[a]	Composition, %		
	Clay M	fly ash	Acidic process water
Type S bricks[b]			
S	100	0	0
Type A bricks[b]			
A1	97.5	2.5	0
A2	95	5	0
A3	90	10	0
A4	80	20	0
A5	60	40	0
Type B bricks[b]			
B1	97.5	0	2.5
B2	95	0	5
B3	90	0	10
B4	80	0	20
B5	60	0	40
Type C bricks[b]			
C1	97.5	2.5	5
C2	95	5	5
C3	90	10	5
C4	80	20	5
C5	60	40	5

[a] Each brick type was produced in batches of 5 identical samples for repeatability and statistical accuracy.
[b] Type S, A and B bricks contained clean water at a ratio of as 10% of the dry solids contained. Type C bricks contained clean water at a ratio of 5% of the dry solids contained since another 5% came from the acidic process water.

A total of 5 identical cylindrical green brick bodies were produced from each coded batch "for leaching, water retention and flexural strength tests"(16 coded sample × 5 each = 80 samples) for assuring repeatability and statistical accuracy. All the brick samples were dried in an oven at 107 °C for a day and fired for 4 h at 1080 °C in accordance with the procedure used in commercial production line.

2.2.2. Determination of water retention capacity

To determine the absorptive capacity of the bricks for water, the brick samples were kept in water for a period of 1 week. The solid/liquid ratio in these tests was always 1/10. Each sample was weighed before and after immersion. The following equation was used to determine water retention capacity:

$$\text{Water retention}\,(\%) = \frac{m_f - m_i}{m_i} \times 100$$

where m_i is the weight of the bricks immediately after firing and m_f is the weight of the same bricks after 1 week of immersion in water. This was based on the Turkish and ASTM standards for testing these bricks [28–30].

2.2.3. Flexural strength measurements

Flexural strength, also known as modulus of rupture or fracture strength, is measured in terms of stress. It is reported as either N/mm^2 or MPa [28–30]. The value represents the highest stress experienced within the material at the moment of its rupture. The apparatus used for this test was Schimadzu AG-1 250 kN model mechanical test setup.

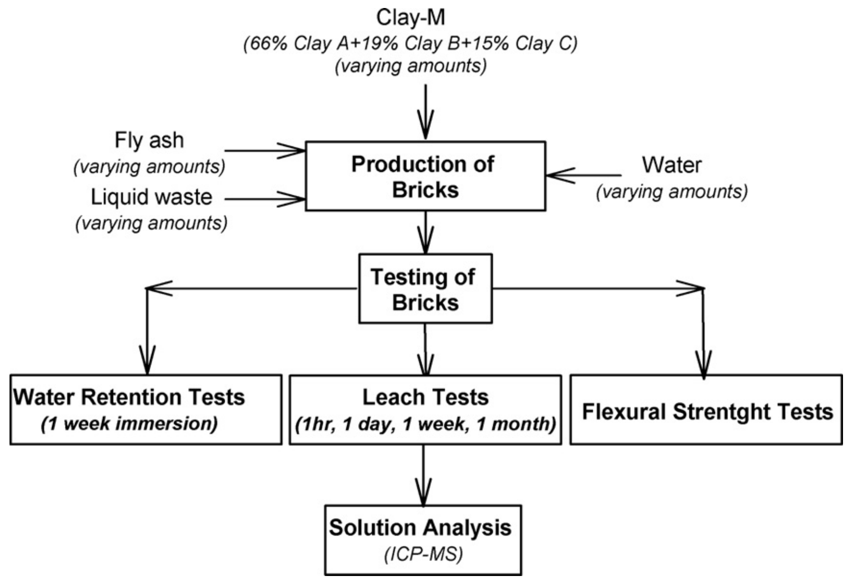

Fig. 1. The flowsheet of the experimental study.

2.2.4. Kinetic leaching tests

The fired brick samples were placed in distilled water at a solid/liquid ratio of 1/10. The bricks were kept in water for 1 h, 1 day, 1 week and 1 month to observe the amounts of metals leaching into solution. At the end of each immersion period, solution samples were obtained using 0.45 μm membrane filters and were analyzed using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS Agilent 7500). A simple flow sheet of the experimental procedure is given in Fig. 1.

3. Results and discussion

3.1. Water absorption capacity and flexural strength

Water absorption capacities and flexural strengths of the brick samples (see Table 3) after firing are given in Table 4.

It can be seen from Fig. 2 that addition of either fly ash or acidic process waste water decreases the water retention capacity of the clay bricks at all ratios when compared to the standard Type S bricks except in the case of 40% fly ash addition alone. The water retention capacity for the bricks containing both fly ash and acidic process waste water is lower than the standard bricks for all ratios, which is quite desirable as far as brick quality is concerned.

The flexural strength measurements are also quite promising. Addition of fly ash alone resulted in stronger bricks for all fly ash ratios such that the flexural strength of 12 MPa for a standard brick increased to around 18–19 MPa when fly ash was added to the brick structure. This may be due to a better binding of the particles in the green body stage due to some pozzolanic reactions between water and alkaline fly ash particles during curing (drying) of the green body. The flexural strength remained nearly unchanged when acidic process waste water was added alone to the brick material, suggesting that there were limited reactions between the clay particles and the waste water. In the case of simultaneous addition of fly ash and acidic process waste water, the flexural strength increased progressively from 12 MPa (for a standard brick) to 15.4 MPa for a brick sample containing 40% fly ash + 5% acidic process waste (Type C5). In short, the water retention capacity and the flexural strength of the bricks showed positive changes with the simultaneous addition of fly ash and acidic process waste water into brick material when compared to the standard bricks composed of

Table 4
Flexural strength and water adsorption capacities of the fired brick samples.

	Water retention capacity, %			Flexural strength, MPa		
	Type A	Type B	Type C	Type A	Type B	Type C
S	9.1	9.1	9.1	12.0	12.0	12.0
1	4.0	10.3	2.2	19.5	11.0	10.2
2	4.4	5.5	4.9	19.1	11.2	12.1
3	7.2	3.6	3.3	18.7	11.3	13.9
4	11.1	2.3	3.3	17.6	12.1	14.4
5	13.7	3.7	6.1	18.6	12.9	15.4

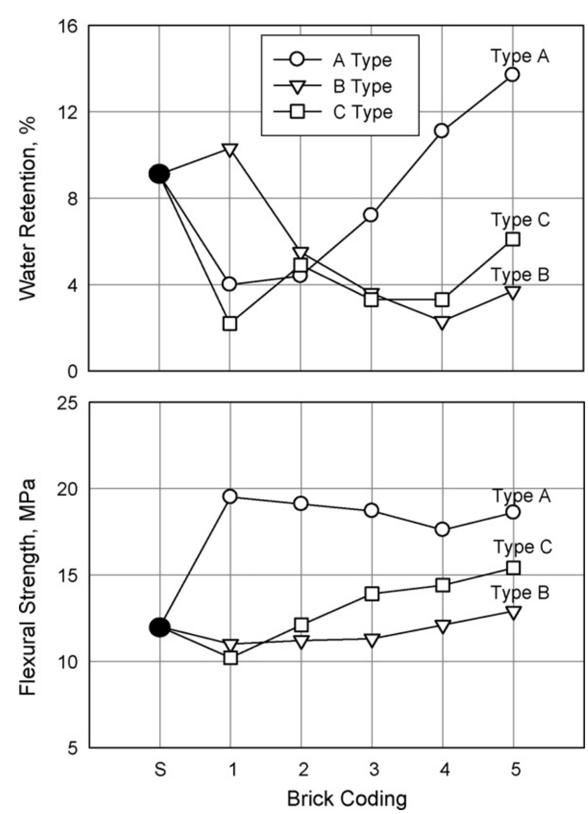

Fig. 2. Water retention capacity and flexural strength as a function of additive amount for various brick samples.

clay alone. These findings are in a good agreement with the findings of other work in the literature [11–27]. Among these studies, for example, Kute and Deodhar [22] showed that the compressive strength of bricks baked at a certain temperature can be improved by 4–5 times for some proportions of fly ash. Still some other work demonstrated that the properties of the test bricks containing fly ash were found to be at least comparable, if not better than, to those of standard test bricks made without fly ash and met the commercial specifications for fired bricks. According to Tutunlu and Atalay [21], the use of fly ash as a raw material for the production of building bricks is not only a viable alternative to clay but also a solution to a difficult and expensive waste disposal problem.

3.2. Leaching behaviour of bricks

Leaching tests were carried out with the experimental bricks to determine the amount of metal leaching from the fired samples. The results of the leaching tests are summarized in Figs. 3 and 4.

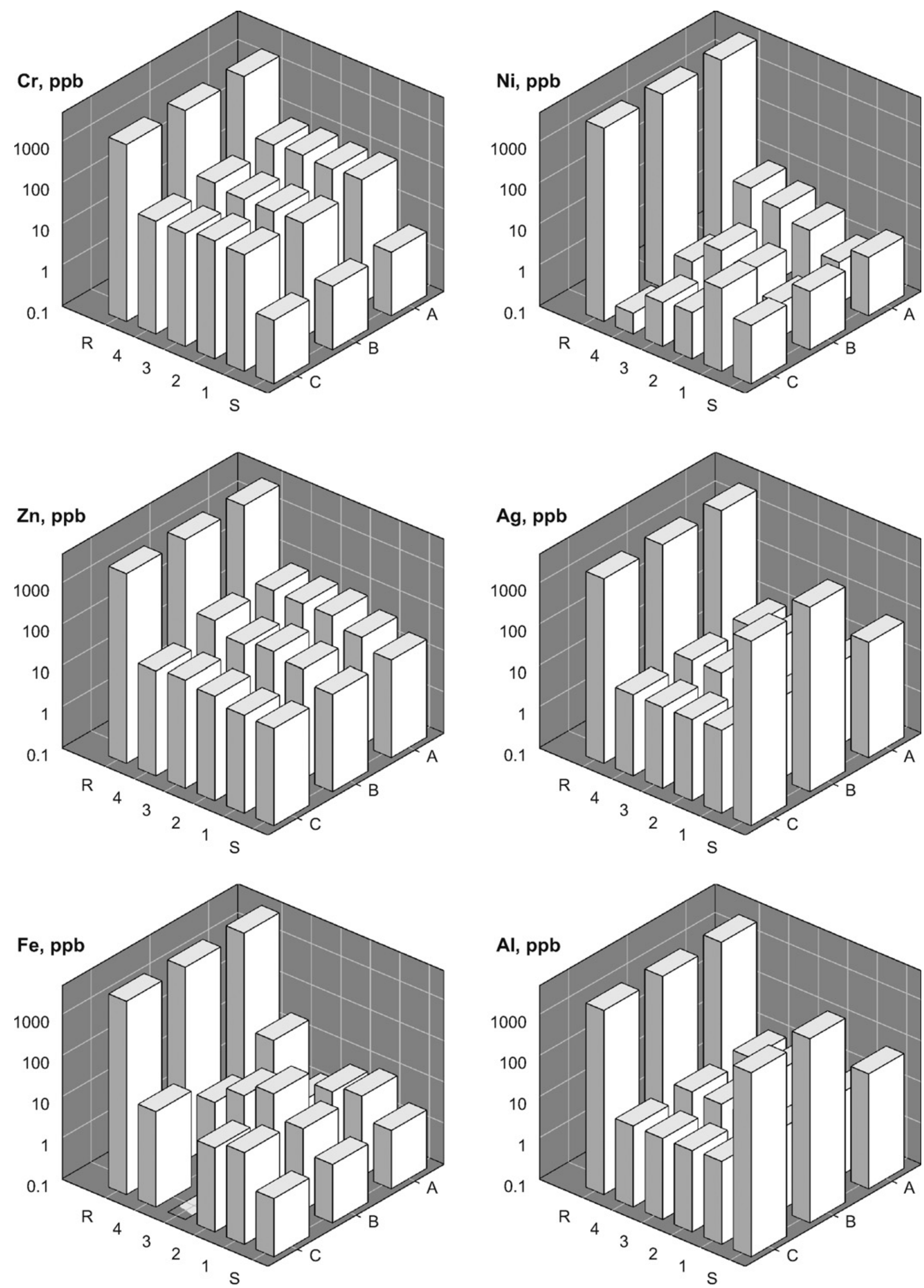

Fig. 3. Cr, Ni, Zn, Ag, Fe and Al metals leached from bricks at the end of 1 month. The *X*-axis denotes the brick types A, B and C while the *Y*-axis denotes the brick coding as S for standard bricks and 1–4 for bricks with different fly ash and acidic process waste water ratios (see Table 3). The code R on the *Y*-axis denotes the discharge standard set by the Turkish Ministry of Environment and Forestry.

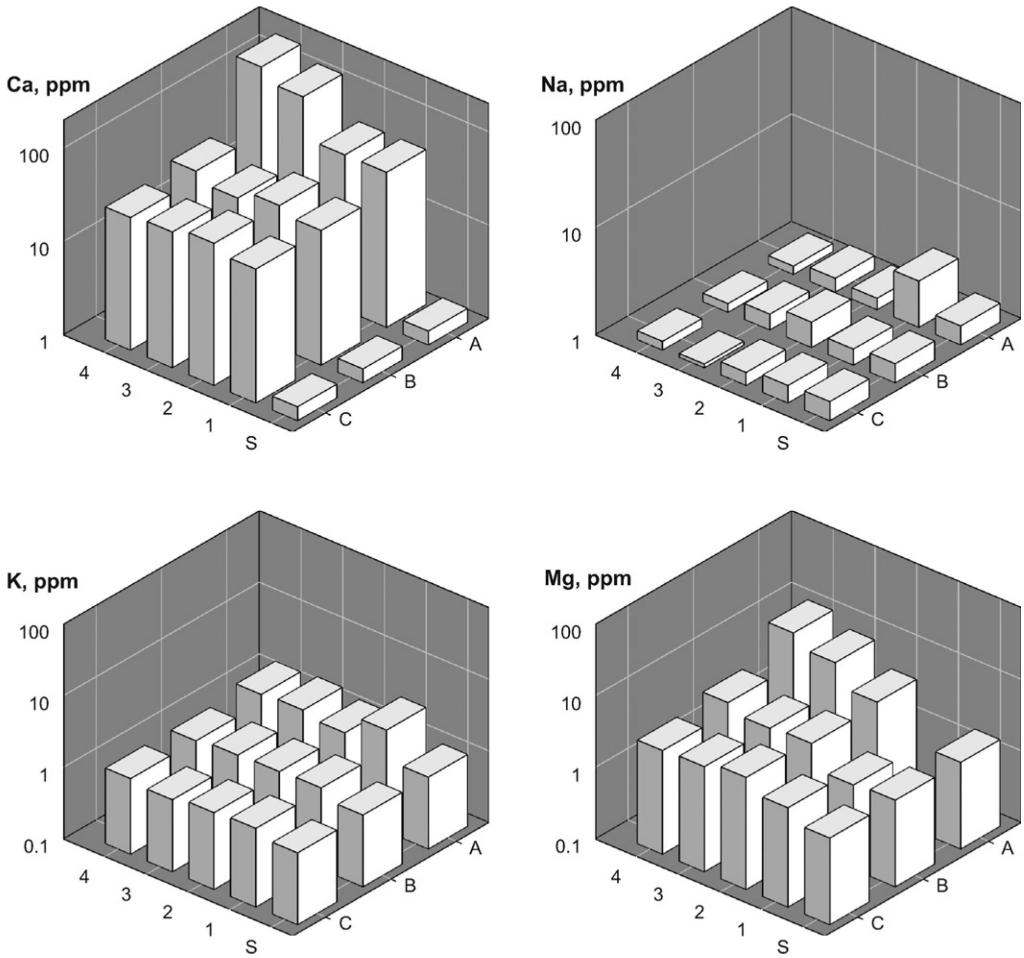

Fig. 4. Ca, Na, K and Mg metals leached from bricks at the end of 1 month. The *X*-axis denotes the brick types A, B and C while the *Y*-axis denotes the brick coding as S for standard bricks and 1–4 for bricks with different fly ash and acidic process waste water ratios (see Table 3). There is no code R on the *Y*-axis for this figure since there are no regulations stated for Ca, Na, K and Mg by the Turkish Ministry of Environment and Forestry.

The ICP-MS analyses of the leach solutions from all the brick types showed that concentrations of metals (such as Mn, Co, Ni, Cu, Tl, Bi, Pb, Cd, and In) and As, in the leach solutions were always below 10 ppb after the 1-month leach period. Hence, only those elements which gave readings above 10 ppb (namely Cr, Ni, Zn, Ag, Fe and Al as heavy metals and Ca, Mg, K and Na as alkaline earth metals) are reported in Figs. 3 and 4.

Figs. 3 and 4 give the leach results for standard Type S bricks in addition to brick types A1–A4, B1–B4 and C1–C4. The bricks A5, B5 and C5 which contained 40% fly ash were excluded from the leaching tests since no additional benefits had been observed with these bricks in terms of water retentions and mechanical strengths. The limiting permissible regulation value for any specific element is also included in Figs. 3 and 4 (as input R in the *Y*-axis) for comparison purposes. These regulation values for the metals reported in these figures are also summarized in Table 5.

Though a detailed discussion can be made for each metal, only a general discussion will be done here for sake of brevity. The bottom line of Figs. 3 and 4 is that leaching of all the metals from all the brick types was found to be below the permissible limiting values set by the Environmental Regulations for waste water. In the case of Cr, Zn and Fe, release of these metals from the bricks which contained ash or process waste water was higher compared to the standard Type S bricks. However, the leached amounts were lower than the permissible limits for these metals for all brick types. Interestingly, for Ni, Al and Ag metals, the leaching from the bricks mixed with fly ash and acidic process waste water was even lower than that observed from the standard bricks.

Though much lower than the allowed discharge limits, the Ca, K and Mg dissolution from the bricks mixed with fly ash and acidic process waste water was higher than the standard bricks. On the other hand, the situation was reverse for Na.

Based on these results, it is clear that metal released into solution from the Type C bricks (which contain both the fly ash and acidic waste additives) is always lower than the permissible values (even less than the standard bricks in several instances) most probably due to fixation reactions developing between the acidic water and the alkaline fly ash. Some studies have suggested physical adsorption of metals onto fly ash surfaces to explain the adsorp-

Table 5
Limiting values set by the Environmental Regulations for waste water [31] for those metals which were detected by ICP-MS to be over 10 ppb in leach solutions.

Heavy metals	Allowable limit, ppm	Alkaline earth metals	Allowable limit, ppm
Cr	2	Ca	NL
Ni	5	Na	NL
Zn	3–5	K	NL
Ag	1–5	Mg	NL
Fe	3–10	pH	6–9
Al	3		

NL: no limit set by the regulations.

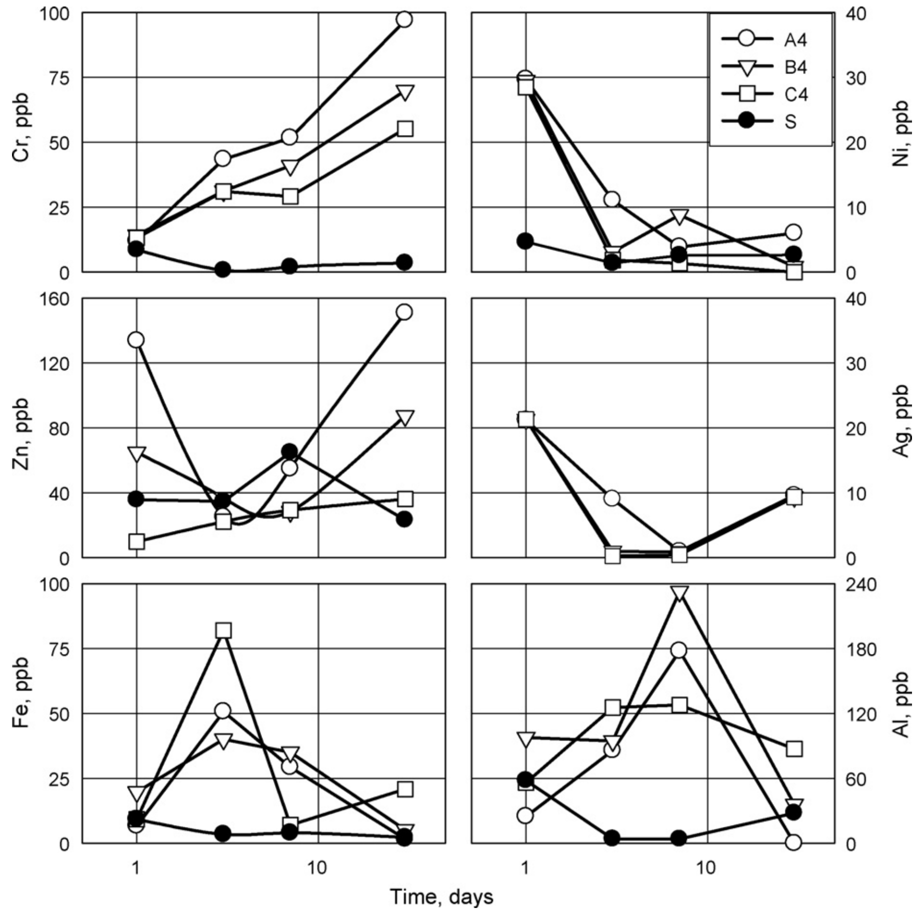

Fig. 5. Cr, Ni, Zn, Ag, Fe and Al metals leached from bricks as a function of time for a period of 1 month. Only the bricks with 20% fly ash content (A4, B4 and C4) are reported here along with the standard bricks (S).

tive behaviour of fly ash [32–34]. Kanungo and Mohapatra [35] proposed a two-step hydrolysis process to explain the decrease of metal ion concentration in solution and relate it to the solubility constants. In addition to the physical adsorption and hydrolysis, Polat et al. [36] suggested surface complexation of the metals to explain the metal adsorption in acidic solutions. Polat et al. also proposed three consecutive processes for the metal removal for different pH regions. According to these researchers, the solution

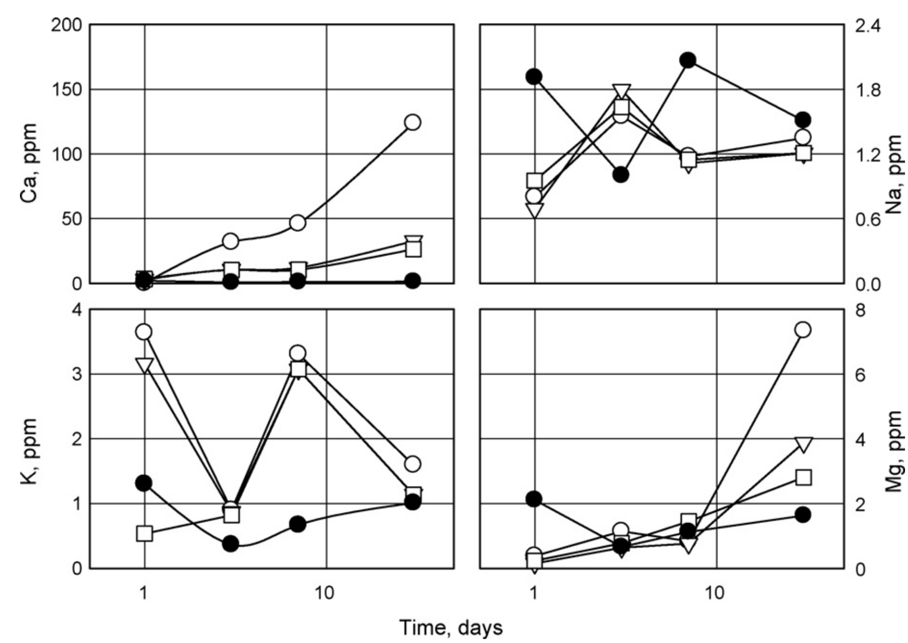

Fig. 6. Ca, Na, K and Mg metals leached from bricks as a function of time for a period of 1 month. Only the bricks with 20% fly ash content (A4, B4 and C4) are reported here along with the standard bricks (S).

$Me^{2+}_{(sol)}$ species may adsorb through coordinative bonding onto the neutral $[S–OH]^0$ sites up to or around the pH_{pzc} of the solid. In the case of the fly ash used in their study, this process took place in a pH region between 3 and 6 and was responsible for significant decreases in metal concentrations. This was followed by simple electrostatic adsorption of the metal cations, $Me^{2+}_{(sol)}$, or the first hydroxo-complexes, $MeOH^{+}_{(sol)}$, after the pH_{pzc} onto a progressively more negatively charged surface. The final stage is simple precipitation, which forms the $Me(OH)_{2(s)}$ species after the hydrolysis pH of each specific metal. According to Polat et al., the coordinative bonding and electrostatic adsorption, which seem to cause significant metal removals from solution, are more favorable environmentally since they take place at lower solution pH levels and are less sensitive to pH changes in solution compared to hydrolysis.

Some more details on metal leaching from fired bricks are presented in Figs. 5 and 6 where the leach data in Figs. 3 and 4 is presented as a function of time for S, A, B and C type bricks for the specific case of 20% fly ash addition. The main conclusion from Fig. 5 is that the Type C bricks where both fly ash and acidic process waste water were simultaneously added to the brick material demonstrate relatively lower dissolution. The even lower dissolution observed with the Type S bricks is because of the fact that the brick clay simply does not contain any of the heavy metals reported in the figures. Also, even though the leached amount of Cr increased with time, it decreased for Ni, Ag, Fe and Al. This suggests a re-precipitation of the latter taking place within the bricks as the leach time progresses.

For the alkaline earth metals, leaching from the bricks seems to increase with time except for K. Especially Ca seems to dissolve quite readily, due to high content of the Ca in the structure of the fly ash originating from the use of lime slurry for sulphur removal in the stack. Such dissolution reactions, however, makes the solution pH move towards neutral values which is desirable for the neutralization of the acidic waste added.

The final pH values of the brick solutions after 1 month of immersion were approximately 6.1 for standard samples, between 6.5 and 9.1 for Type A bricks, between 5.0 and 6.3 for Type B bricks and between 5.9 and 8.5 for Type C bricks. It can be seen that the bricks which contained both the fly ash and the acidic waste showed neutral solutions after long immersion times, perfectly within the permissible environmental limit of 6–9.

Again, it should be stated that all the alkaline earth metals dissolved below the permissible environmental limits after a leach time of 1 month.

4. Conclusions

The purpose of this study was to use a solid (fly ash) and a liquid (acidic tanning factory process water) waste as additives in the production of construction bricks in order to eliminate these wastes by permanently fixing the heavy metals in the structure of the fired bricks. Water retention, flexural strength and leaching tests were carried out with experimental bricks with and without the additives under several conditions.

The results show clearly that simultaneous addition of fly ash and acidic waste into clay increases the mechanical strength of the fired bricks while lowering the water retention capacity compared to those observed with the standard bricks which contained no additives. More importantly, the bricks from clay–fly ash–acidic waste water mixtures do not release any metals above the permissible limits set by the regulations. The metal release for simultaneous addition of fly ash and acidic waste water is lowest in several cases most probably due to pozzolanic reactions between the alkaline ash and acidic waste water irreversibly fixing the metals in the brick structure under normal exposure conditions.

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