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Thermal treatment of toxic metals of industrial hazardous wastes with fly ash and clay

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Abstract

Waste generated from galvanizing and metal finishing processes is considered to be a hazardous due to the presence of toxic metals like Pb, Cu, Cr, Zn, etc. Thermal treatment of such types of wastes in the presence of clay and fly ash can immobilizes their toxic metals to a maximum level. After treatment solidified mass can be utilized in construction or disposed off through land fillings without susceptibility of re-mobilization of toxic metals. In the present investigation locally available clay and fly ash of particular thermal power plant were used as additives for thermal treatment of both of the wastes in their different proportions at 850, 900 and 950 °C. Observed results indicated that heating temperature to be a key factor in the immobilization of toxic metals of the waste. It was noticed that the leachability of metals of the waste reduces to a negligible level after heating at 950 °C. Thermally treated solidified specimen of 10% waste and remaining clay have shown comparatively a higher compressive strength than clay fired bricks used in building construction. Though, thermally heated specimens made of galvanizing waste have shown much better strength than specimen made of metal finishing waste. The lechability of toxic metals like Cr, Cu, Pb and Zn became far below from their regulatory threshold after heating at 950 °C. Addition of fly ash did not show any improvement either in engineering property or in leachability of metals from the solidified mass. X-ray diffraction (XRD) analysis of the solidified product confirmed the presence of mixed phases of oxides of metals.

Keywords: Galvanizing waste; Clay; Fly ash; Thermal treatment; Toxic metals

1. Introduction

Immobilization of organic or inorganic contaminates of an industrial waste is essential prior to land disposal or utilization of waste in usable purposes. Otherwise, physical and chemical changes due to weathering or other effects can starts the release of contaminates from the waste in an unacceptable limit. This may results contamination of the soil, surface and ground water. In case of inorganic waste, thermal treatment may solves two problem in one step: (i) immobilization of toxic elements and (ii) solidified mass can be utilized as building materials. Thermal treatment approaches like vitrification, sintering, bricks firing, etc. are being employed now-a-days for the stabilization of heavy metals bearing industrial and sewage sludge. Among these, bricks firing process is considered to be promising one because of the involvement of low cost and ease in accessibility. The stabilized mass obtained by this process could be utilized as building materials or disposed off through land fillings without susceptibility of leachability of toxic metals. Use of this approach of treatment is reported for the conversion of industrial sludge as the bricks or tile additive [1–5].

Earlier studies have shown that mobility of many toxic metals decreases with the increase of thermal treatment temperature [6–8]. In case of metal finishing and galvanizing wastes which are mainly contains heavy metals in their oxide and oxyhydroxide forms, thermal treatment may facilitates their involvement with other component of additives. During heating in the presence of clay/and or fly ash, immobilization of toxic metals of the waste could be achieved by the formation of stable compound with Si or Al of clay or other constituent of fly ash. Addition of an appropriate amount of clay and/or fly ash can also increases the engineering properties of the solidified product. However, no reports is available in the literature which could discuss a details understanding of thermal immobilization of toxic metals

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of metal finishing and galvanizing wastes in the presence of clay and fly ash. Secondly, measurement of engineering properties of stabilized specimen is equally important for the development of a stable product that can be utilized in construction purposes.

In this study specimens made of waste, clay and fly ash in their varying ratios were heated at 850 °C and above temperatures. Leachability of the toxic metals from the solidified product was compared with their leachability before and after thermal treatment. Identification of phases formed after solidification was analyzed by X-ray diffraction (XRD) while toxicity characteristics leaching procedure (TCLP) was employed for the determination of leachability of metals. The compressive strength, water absorption, bulk density, firing shrinkages of the thermally treated specimens was measured to evaluate their stability and possible uses as building product.

2. Experimental procedure

2.1. Materials

Semi dried metal finishing (designated as MF waste) sludge (moisture ∼ 35%) and galvanizing (designated as GI waste) sludge (moisture ∼ 20%) was collected from local industries. Both sludges were dried in oven at 110 °C till the removal of moisture. The density of dried sludge was found to be 0.75 and 1.12 g cm^{-3}, respectively for GI and MF waste. Qualitative analysis of the wastes were performed by Jeol, Japan make JSM 5600 SEM attached with link ISIS 300, Oxford, UK made Energy Dispersive X-ray (EDAX) spectrometer. EDAX spectrum as demonstrated in Figs. 1 and 2, pertain the presence of Ca, Cl, Si, Mg, etc. along with Zn, Fe in the waste. Some of the metals like Mn, Pb, Cu, Cr, Ni, Co, etc., could not be detected in EDAX analysis due to their low percentage. Therefore, quantitative analysis of above metals along with identified Fe, Zn of the wastes was made by Atomic Absorption Spectrophotometer (GBC Australia make, model 902) at their specific wavelength. The quantitative analysis of the wastes is given in Table 1. The compositional analyses of local clay and fly ash of a particular thermal power station used as an additive with waste in the thermal treatment are presented in Table 2.

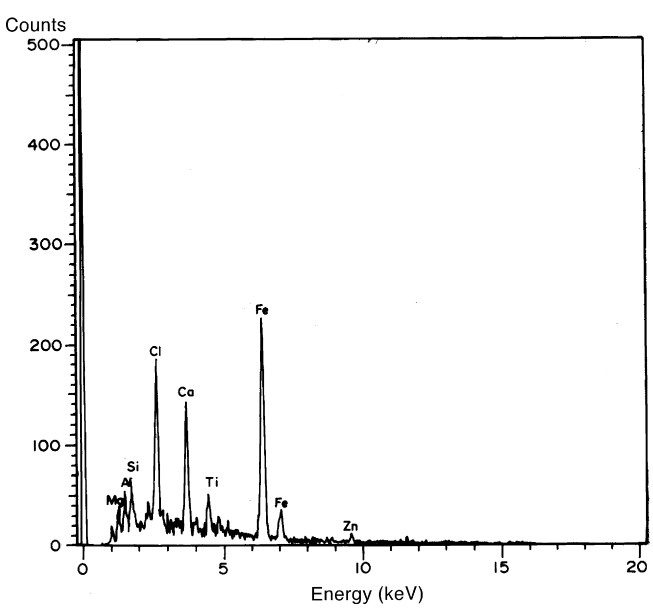

Fig. 1. EDAX spectrum of the metal finishing waste.

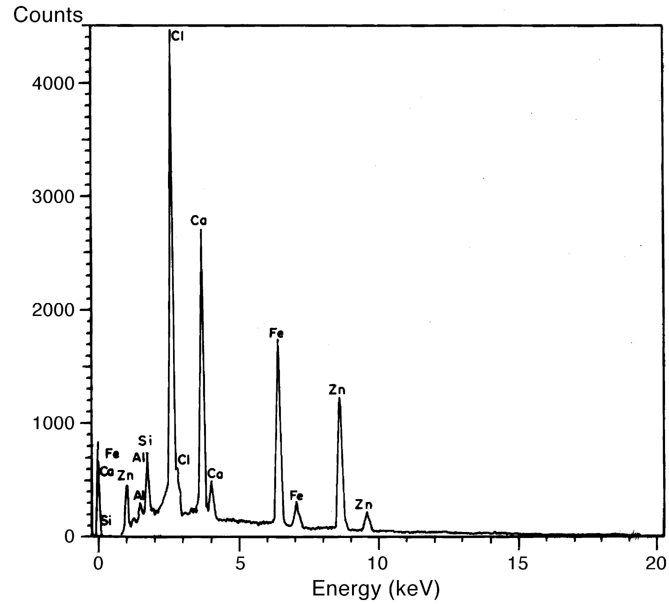

Fig. 2. EDAX spectrum of galvanizing waste.

Table 1
Presence of heavy metals in metal finishing and galvanizing sludge

Element	Metal finishing waste	Galvanizing waste
Iron	15.9%	22.6%
Copper	0.45%	Nil
Zinc	2.6%	13.6%
Chromium	319 ppm	Nil
Manganese	302 ppm	407 ppm
Lead	160 ppm	680 ppm
Nickel	451 ppm	Nil
Cobalt	60 ppm	Nil
Cadmium	Nil	Nil

2.2. Method

Dried sludge, clay and fly ash were grinded in identical size of ∼200 μm and mixed homogeneously in their differ-

Table 2
Molecular composition of clay and fly ash used in thermal treatment

Constituents	Clay	Fly ash
Fe_2O_3	5.82	5.6
Al_2O_3	18.33	24.0
SiO_2	63.38	58.2
CaO	1.66	2.2
MgO	1.37	1.8
Na_2O	0.68	2.6
K_2O	0.65	0.8

ent proportions for making brick specimen (100 mm length, 50 mm width and 50 mm thickness) by compressive molding technique. Twelve to thirteen percent of water was added in mixture before applying 150 kg cm^{-2} pressure by Shimadzu, Japan make (model UH-200A) machine. After molding, specimens were removed from the machine and kept for initial settling in air for 24 h. Then, the specimens were dried at 110–115 °C in oven till a constant weight occurred. Thereafter, specimens were kept for heating in muffle furnace at 850, 900 and 950 °C for 2 h. After furnace cooling heated specimens were removed from the furnace and subjected for important test like compressive strength, water absorption, firing shrinkage, bulk density measurements as per procedure of test of burnt clay building bricks [10]. The surface morphology of the thermally heated specimens were seen by scanning electron microscopy (Jeol, Japan make JSM 5600) while phase analysis of the specimens were performed by X-ray diffraction (Bruker aXS, model D8 Advance, Japan) by utilizing Cu Kα radiation at 20.500 wavelength. The identification of phases formed after heating of the specimens, was made by software named as EVA attached with XRD machine.

2.3. TCLP test

TCLP test was carried out for the determination of leachability of heavy metals present in the thermally treated specimens. In this test, 25 g of grinded solidified specimen (size ∼ 0.9 mm) was mixed in extraction liquid of acetic acid and sodium hydroxide of pH 4.93 as per US EPA [11]. The details of procedure employed in the TCLP test is given elsewhere [11,12]. Hexavalent chromium [Cr(VI)] of the TCLP leachates was determined by diphenyl carbohydrazide method [13,14] utilizing microprocessor based spectrophotometer (Hach DR-2000, USA). Total chromium and remaining metals of the leachates were analyzed by AAS.

3. Results and discussion

3.1. Compressive strength

Measurement of compressive strength is essential in order to assure the engineering quality of solidified mass suitable in building construction [15]. The compressive test results of heated specimens at 850, 900 and 950 °C are shown in Figs. 3 and 4, respectively, for MF and GI waste bearing specimens. Results show that strength is mainly dependent on the waste content, clay ratio and heating temperature. With the increase of heating temperature, compressive strength increases. In the presence of 10% waste with remaining clay a maximum strength of around 110 and 250 kg cm^{-2} were measured for MF and GI waste bearing specimens, respectively. This range of strength is higher than minimum strength required (75 kg cm^{-2}) for normal clay bricks used in building construction. The strength of the specimen made of clay alone (same clay used in the present investigation as additive with waste), was found to be 105 kg cm^{-2} Observed data indicate that addition of fly ash did not show any improvement in the strength of

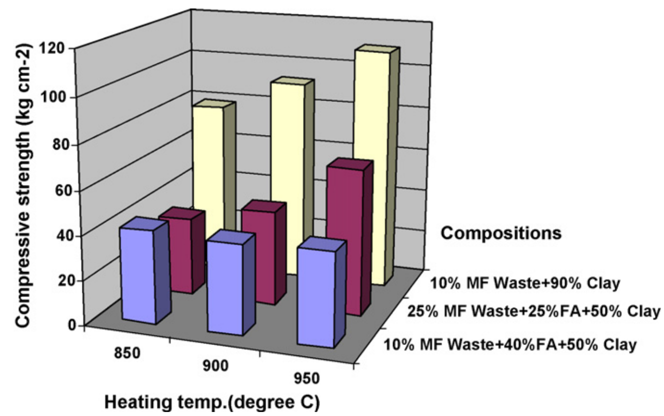

Fig. 3. Compressive strength of thermally treated specimens of metal finishing waste in its different compositions with clay and fly ash.

the specimens. In fact, addition of fly ash decreases the compressive strength as it reduced to more than 60% in the presence of 40% fly ash with 10% waste and 50% clay as compared to strength occurred for specimen of 10% waste and remaining clay.

The effect of waste addition on the compressive strength of solidified specimens was evaluated separately. For carrying this test an incremental addition of 5% waste in the clay was made in specimen's preparation. The prepared specimens were heated at 950 °C for 2 h. Results of compressive strength was plotted against waste content, is presented in Fig. 5 for both of the waste bearing specimens. As shown, the compressive strength occurs near to 220 and 105 kg cm^{-2}, respectively, for GI and MF bearing waste in the presence of their 15% weight with clay. After this addition, the compressive strength decreases linearly and became less than 100 and 40 kg cm^{-2}, respectively, in 30% GI and MF waste bearing specimens. Interestingly, more than two times higher compressive strength was observed for GI waste bearing specimen (Fig. 6) as compared to MF bearing specimen. This observation reveals that GI waste is more suitable for making of clay fired bricks.

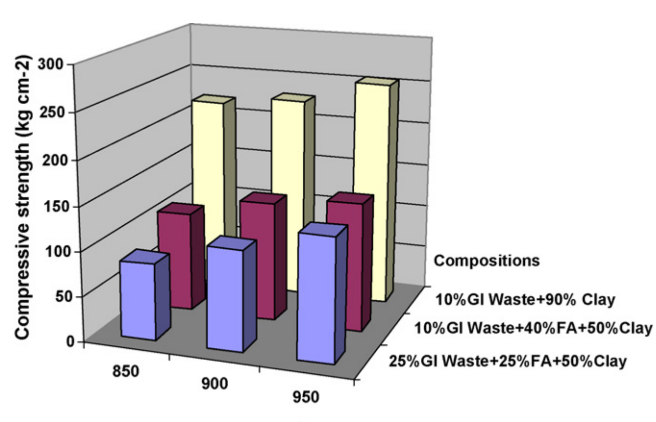

Fig. 4. Compressive strength of thermally treated specimens of galvanizing waste in its different compositions with clay and fly ash.

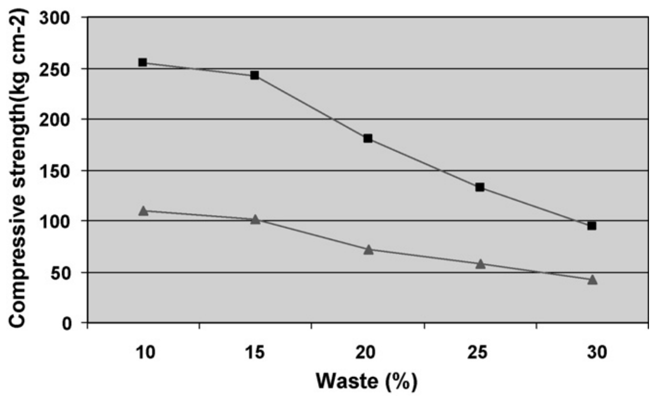

Fig. 5. Effect of metal finishing (▲) and galvanizing (■) waste addition with clay on the compressive strength of thermally treated specimens at 950 °C.

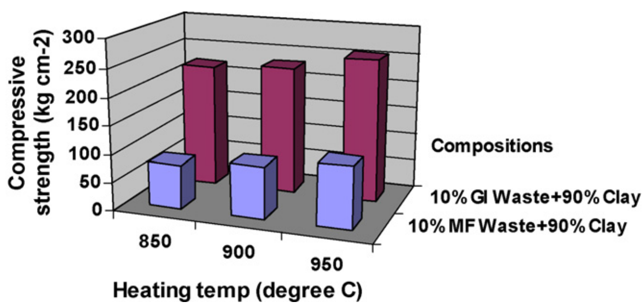

Fig. 6. Comparative compressive strength of thermally treated 10% galvanizing and metal finishing waste bearing specimens at different temperatures.

3.2. Water absorption

Absorption of water by clay fired bricks is another important property, determines the stability and durability of clay fired bricks. Less water absorption indicates the presence of more durability in the solidified product. Absorption of more water can increase the leachability of metals due to increase of solubility of metal compounds in the solidified matrix. Water absorption measured for solidified specimens of three compositions for both types of waste bearing specimens heated at 850, 900 and 950 °C, are given in Table 3. From the result one can see that water absorption decreases with the increase of heating temperature. Around 15 and 16% of water absorption was measured for specimens containing 10% GI and MF waste, respectively, in the presence of clay heated at 950 °C. This percentage of water absorption comes under the limit of normal brick used as building materials.

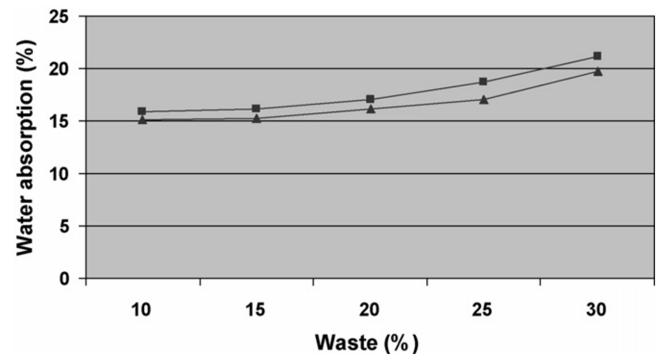

Fig. 7. Effect of metal finishing (■) and galvanizing (▲) waste addition with clay on the water absorption of thermally treated specimens at 950 °C.

The effect of waste content addition with clay on the water absorption rate of heated specimens at 950 °C, was measured. Observed results as demonstrated in Fig. 7 show that water absorption increases with the increase of waste content. After addition of 15% waste, the water absorption becomes more than 17 and 18%, respectively, for GI and MF waste bearing specimens. Surface morphology of the heated specimens of different composition of waste and clay was also seen by SEM. SEM photomicrographs taken for thermally treated specimens of MF and GI containing waste with clay are depicted in Figs. 8 and 9, respectively. As compared to 15% MF waste bearing specimen (Fig. 8a), one can see an increased number of porosity at the surface of 25% waste containing specimen (Fig. 8b). Similarly, numbers of pore on the surface of 25% GI bearing specimen (Fig. 9b) increases as compared to its 15% GI waste bearing specimen (Fig. 9a). The other interesting observation is the appearance of cracks on the surface of MF waste bearing specimens (Fig. 8a and b). However, no such cracks are appeared on the surface of GI waste bearing specimens (Fig. 9a) and thermally treated mass looks more compact in nature. Based on the surface morphology of thermally treated specimen it appears that increases of waste in the mixture increases the porosity or voids in the solidified product. This leads a decrease in compressive strength and increases in water absorption ratio. Presents results are in good agreement with the results observed related to compressive strength measurement. Measurement of less water absorption in GI bearing specimen as compared to MF bearing specimen suggest the presence of more cohesiveness in the solidified matrix.

Table 3
Observed water absorption data for heat treated specimens at 850, 900 and 950 °C of different compositions in the presence of galvanizing and metal finishing wastes

	MF waste bearings specimens			GI waste bearing specimens		
	850 °C	900 °C	950 °C	850 °C	900 °C	950 °C
Water absorption (%)						
10% waste + 90% clay	15.9	15.8	15.5	16.5	16.2	15.1
10% waste + 40% FA + 50% clay	21.1	20.8	20.4	17.5	17.1	16.1
25% waste + 25% FA + 50% clay	21.7	21.4	21.0	21.8	21.2	19.6

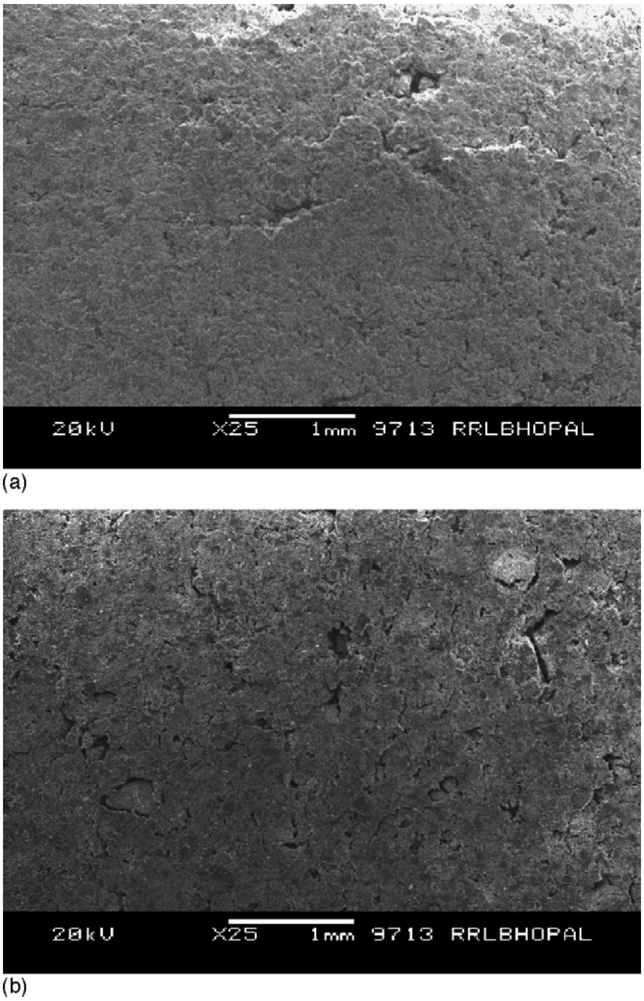

Fig. 8. Surface morphologies of 15% MF waste with clay (a) and 25% MF waste with clay (b) bearing thermally treated specimen at 950 °C.

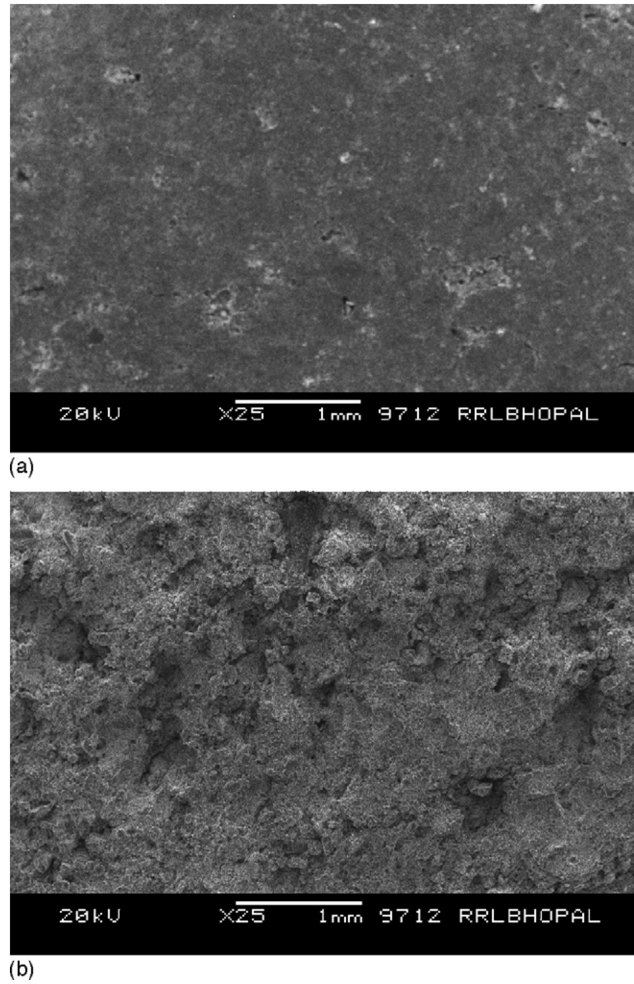

Fig. 9. Surface morphologies of 15% GI waste with clay (a) and 25% GI waste with clay (b) bearing thermally treated specimen at 950 °C.

3.3. Firing shrinkage and density

Firing shrinkage and density of brick are equally important in assuring of their quality. Generally, a good quality of brick posses a maximum firing shrinkage of less than 8% and density in the range of 1.8–2 g cm^{-3} [15]. The firing shrinkage and density measured for both types of specimen of composition: (i) 10% waste + 90% clay, (ii) 25% waste + 25% fly ash + 50% clay and (iii) 10% waste + 40% fly ash + 50% clay after firing at 950 °C, are summarized in Table 4. As shown the shrinkage percentage increases with the increase of waste percentage and bulk density of specimen decreases with the decrease of waste percentages. In comparison to MF waste bearing specimen, GI bearing specimen show less water shrinkage and higher bulk density. Present observations are in similar line to quantity of water absorbed as discussed above. Because the quantity of absorption of water by heated specimens is closely related to presence of porosity or cavities in the matrix. If cavities or porosity are more in the matrix, specimens will exhibit less density and absorb more water.

3.4. TCLP test

TCLP was adapted by the U.S. for the determination of mobility of organic and inorganic contaminants under the Hazardous and Solid Waste Amendment in 1984 [11]. This test is a reg-

Table 4
Observed firing shrinkages and density of specimens heated at 950 °C for 2 h of different compositions in the presence of galvanizing and metal finishing wastes

	MFW bearings specimens		GI bearing specimens	
	Firing shrinkages (%)	Density (g cm^{-3})	Firing shrinkages (%)	Density (g cm^{-3})
10% waste + 90% clay	7.2	1.74	6.4	1.83
10% waste + 40% FA + 50% clay	8.6	1.61	7.9	1.65
25% waste + 25% FA + 50% clay	9.7	1.58	8.9	1.60

Table 5
Analysis of Cr, Cu, Zn and Pb as main toxic elements present in TCLP leachates of studied wastes

	Metal finishing waste				Galvanizing waste			
	Cu	Zn	Pb	Cr	Cu	Zn	Pb	Cr
TCLP leachate (mg l^{-1})	2.45	36	2.1	0.32	Nil	165	2.4	Nil
Regulatory threshold (mg l^{-1})	15.0	25.0	5.0	5.0	15.0	25.0	5.0	5.0

ulatory test, determines whether a particular waste meets the applicable technology based treatment standard for land fillings. TCLP results for main toxic metals of the studies waste namely Cu, Zn, Pb and Cr obtained from waste alone are given in Table 5. Presence of 36 and 165 mg l^{-1} Zn in the leachates of MF and GI waste, respectively, which are more than threshold value of Zn as per US EPA [9] confirm their hazardous characteristic. If consider toxicity due to Zn, GI waste appears to be a more hazardous. In addition, Cu and Cr in leachates of MF waste and Pb in the leachates of GI waste were found. Though their concentration was less than regulatory threshold concentrations as per US EPA [9] (Table 5). 8.2 and 12.5 mg l^{-1} Fe was also found in the leachates of MF and GI waste, respectively. Whereas very less concentration of Mn as 0.16 and 0.28 mg l^{-1} in MF and GI waste, respectively, was analyzed in their respective leachates. Other heavy metals like Co, Ni and Cd did not detect in the TCLP leachates of the waste. Presence of Zn along with highly toxic Cu and Pb in the TCLP leachates of MF waste and Pb in the TCLP leachates of GI waste suggests their immobilization is necessary prior to the disposal of waste or utilization in usable purposes.

TCLP results of Cu, Pb and Zn obtained from solidified specimens at 850, 900 and 950 °C containing 10% MF and remaining clay are compared in bar chart (Fig. 10). Results show that the leachability of Pb reduces with the increase of heating temperature and became very less at 950 °C (∼0.10 mg l^{-1}). It is reported that PbO reacts with various sorbent flakes at a temperature greater than 700 °C and make a complex compound $MO{\cdot}Al_2O_3$, $MO{\cdot}2SiO_2$, etc. where M represent for Pb [7,8]. Involvement of PbO with oxides of Al and Si of clay is expected in the present case. While a slight increase of lechability of Cu was noticed with the increase of heating temperature. This observation is somewhat surprising because a decrease in leachability of Cu is reported with the increase of thermal treatment temperature of Cu contaminated soil [7]. The leachability of Zn occurs below to their threshold concentration even at 850 °C and became less than 5.8 mg l^{-1} after heating at 950 °C. In case of 10% GI waste and remaining clay containing heated specimen at 850 °C, the leachability of Zn occurred much more (110 mg l^{-1}) than threshold concentration (25 mg l^{-1}). However, its concentration in the leachates decreases considerably after heating at 900 °C and occurred within the threshold concentration after heating at 950 °C (Fig. 11). Vaporization of Zn bearing compounds of the waste at 850 °C and their apparent accumulation in the vacant site of the solidified matrix is seem to be the reason for the analysis of its somewhat high concentration. Vaporization of Zn of electroplating waste up to 600–900 °C is reported [16]. Heating at 900 °C, vaporized zinc presumably combines with other component of mixture and attains nearly an immobilization state.

A small concentration of chromium as 0.32 (mg l^{-1}) was estimated as total Cr in the leachates of MF waste alone (Table 5). After carrying analysis for hexavalent chromium nearly 0.27 mg l^{-1} was found as Cr(VI) in the leachates. Interestingly, concentration of Cr(VI) became nearly 10 times higher (∼02.6 mg l^{-1}) in the TCLP leachates of 10% MF waste bearing specimens heated at 850 °C. After heating at 900 °C its concentration in the TCLP leachates reduced significantly (∼1.30 mg l^{-1}) the occurrence of re-oxidation reaction of Cr(III) to Cr(VI) at 600–900 °C [9] is attributed to be the reason for observation of its high concentration in the TCLP leachates of

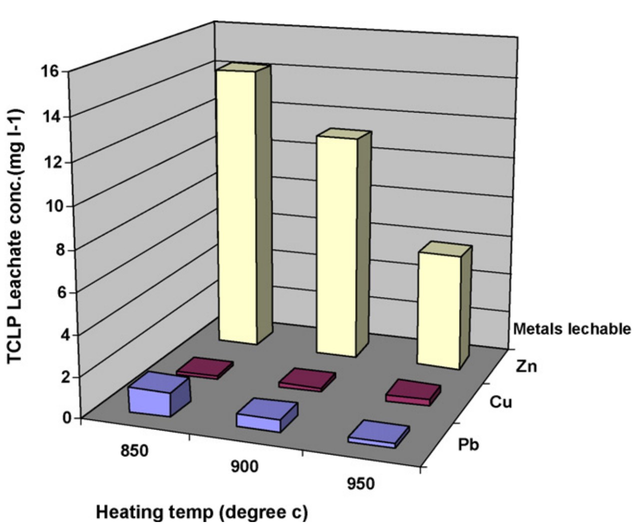

Fig. 10. Leaching concentration of Cu, Pb and Zn in TCLP leachates of thermally treated specimen of various compositions containing metal finishing waste, fly ash and clay at different temperatures.

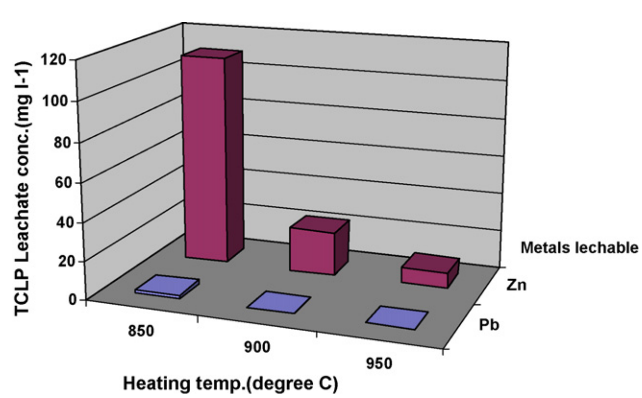

Fig. 11. Leaching concentration of Pb and Zn in TCLP leachates of thermally treated specimen of various compositions containing galvanizing waste, fly ash and clay at different temperatures.

Table 6
Analysis of Pb, Zn and Cu in TCLP leachates of specimens of metal finishing waste in different compositions heated at 850, 900 and 950 °C

	850 °C			900 °C			950 °C		
	Cu[a]	Zn[a]	Pb[a]	Cu[a]	Zn[a]	Pb[a]	Cu[a]	Zn[a]	Pb[a]
Leachate ($mg\,l^{-1}$) in 10% waste + 90% clay	0.12	14.2	1.2	0.23	11.2	0.62	0.31	5.8	0.22
Leachate ($mg\,l^{-1}$) in 10% waste + 40% fly ash + 50% clay	0.21	15.6	1.3	0.29	12.1	0.79	0.42	5.2	0.27
Leachate ($mg\,l^{-1}$) in 25% waste + 25% fly ash + 50% clay	0.46	31.6	3.3	0.54	18.2	1.6	0.67	11.5	0.46

[a] Cu, Zn and Pb are leachable metals.

Table 7
Analysis of Zn and Pb in TCLP leachates of specimens of galvanizing waste in different compositions heated at 850, 900 and 950 °C

	850 °C		900 °C		950 °C	
	Zn[a]	Pb[a]	Zn[a]	Pb[a]	Zn[a]	Pb[a]
Leachate ($mg\,l^{-1}$) in 10% waste + 90% clay	110	2.1	23	0.24	8.4	0.14
Leachate ($mg\,l^{-1}$) in 10% waste + 40% fly ash + 50% clay	108	2.3	28.6	0.22	8.2	0.17
Leachate ($mg\,l^{-1}$) in 25% waste + 25% fly ash + 50% clay	212	5.2	47	0.38	14.2	0.29

[a] Zn and Pb are leachable metals.

specimens heated at 850 and 900 °C. Due to this reaction some of the chromium present in the waste as Cr(III) get oxidized to Cr(VI) at 850 °C (Table 4) which increases the concentration of Cr(VI) in the TCLP leachates. The concentration of Cr(VI) in the TCLP leachates became quite low (<0.12 $mg\,l^{-1}$) when specimens of above composition were heated at 950 °C. These observations confirmed the non-existence of re-oxidation reaction of chromium at 950 °C. Possibility of vaporization of Cr compounds of the waste during heating at 800–900 °C could be ruled out as no such vaporization of Cr of electroplating waste is reported [9,16].

Addition of fly ash does not make any improvement in the decrease of the leaching of above metals. The leachability of above metals in the presence and absence of fly ash is summarized in Tables 6 and 7 for MF and GI waste bearing thermally treated specimens, respectively. From the leachability data, one can see that fly ash addition does not make any noticeable decrease in the concentration of metals in the TCLP leachates. On the other side, clay addition enhances the binding of metals in the solidified matrix. Presence of silica and alumina (known as good solvents) in the clay is likely to improve the adsorption of various constituents of the waste. This ultimately responsible for their immobilization after thermal treatment.

3.5. X-ray diffraction analysis

X-ray diffraction analysis results of 10% MF and GI waste containing clay heated specimens at 950 °C, are presented in Figs. 12 and 13, respectively. In both types of specimen F_2O_3, SiO_2, $CaSiO_3$, $CaO_5Al_2O_3$ constituents are identified as the main phases. In case of GI bearing solidified specimen a mixed oxide phase of Zn, Al and Fe was identified. As Zn present in the free state gets involved with Al and Fe and make their stable oxide after heating at 950 °C. This was the reason that a less leachability of Zn was measured in the TCLP leachates of 10% GI waste bearing solidified specimen heated at 950 °C. In both of the waste bearing specimens no XRD peaks were iden-

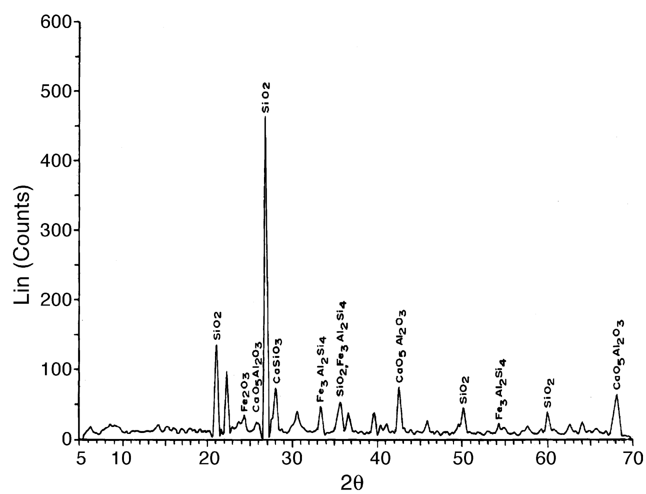

Fig. 12. XRD spectrum of thermally treated specimen containing 10% metal finishing waste and remaining clay at 950 °C.

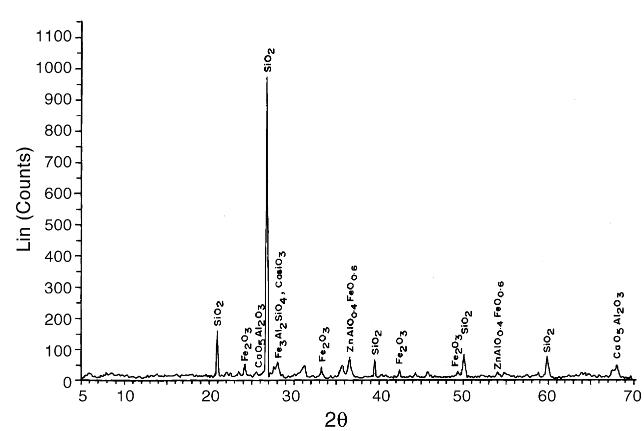

Fig. 13. XRD spectrum of thermally treated specimen containing 10% galvanizing waste and remaining clay at 950 °C.

tified for the presence of mixed phases of Cu, Pb or any other metals. Presence of their small concentration in the specimen is presumably the reason for absence of their XRD peaks in the spectrum. To know whether all the Zn or Cu or Pb of the waste after heating get involved with other components of the mixture or present partially in their free state in the cavities and voids of solidified matrix, further investigations based on in situ analysis during thermal treatment of the specimens could be carried out.

4. Conclusions

Observed data indicate that both, MF and GI waste in their 10–15 wt.% can be stabilized into a stable product after heating at 950 °C with clay. The compressive strength, water absorption, firing shrinkages, bulk density of thermally treated specimens occurs within the specified standard of clay fired bricks used in building construction. In comparison to MF, GI waste bearing thermally treated specimens show a superior quality of brick. Leachability measurement of the thermally treated specimens at 950 °C demonstrates for nearly a complete immobilization of toxic metals of the waste present in the mixture.

Increase of thermal treatment temperature from 850 to 950 °C of the specimens consisting of waste of above percentages with remaining clay improves their engineering properties substantially without showing any noticeable cracks at the surface of heated specimens. However, addition of fly ash in the mixture does not make any improvement either in engineering properties or in the immobilization of toxic metals of the waste. In fact, its addition decreases the compressive strength and increases the water absorption of the thermally treated specimen.

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