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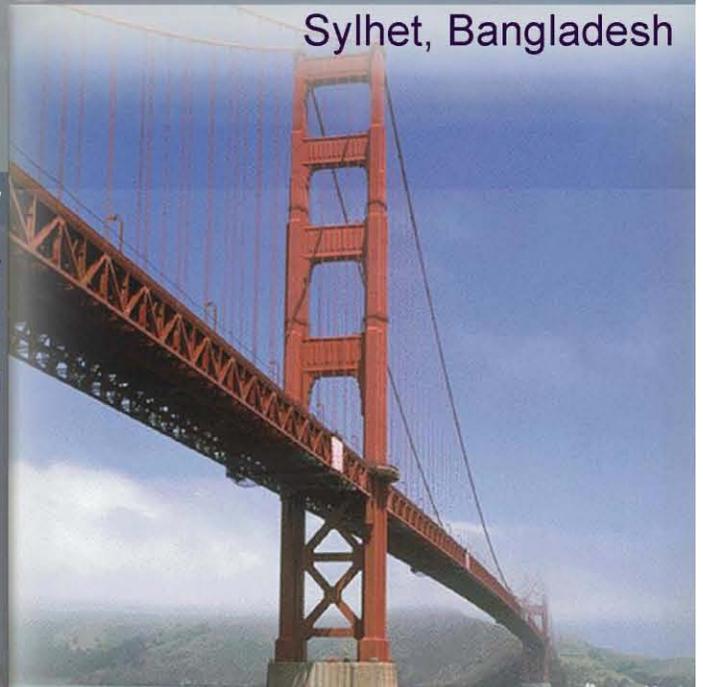
**ICETCESD-2012**



10-12 March 2012  
Sylhet, Bangladesh

Edited by

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Department of Civil and Environmental Engineering  
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## POTENTIALITY OF ELECTROCOAGULATED METAL HYDROXIDE SLUDGE (EMHS) FROM TEXTILE INDUSTRY IN MANUFACTURING OF BUILDING MATERIALS

Tanveer Mehedi Adyel<sup>1</sup>, Syed Hafizur Rahman<sup>1</sup>, S.M. Nazrul Islam<sup>1</sup>, Hossain Md. Sayem<sup>2</sup>, Mala Khan<sup>3</sup>, Md. Abdul Gafur<sup>4</sup>, Mohammad Moniruz Zaman<sup>5</sup> and Md. Mahbubur Rahman Bhuiyan<sup>6</sup>

**ABSTRACT:** Present work was conducted to investigate the reuse feasibility of Electrocoagulated Metal Hydroxide Sludge (EMHS) from textile industry in manufacturing of building blocks. Geo-engineering, elemental, thermal profile and surface microstructure of EMHS were investigated using British Standard 1377, Energy Dispersive X-ray Fluorescence (EDXRF), Thermogravimetric/ Differential Thermal Analyzer (TG/DTA) and Scanning Electron Microscope (SEM), respectively. Different batches of building blocks were prepared using up to 50% EMHS in soil and fired at different temperatures. EMHS proportion and firing temperature were the two key factors determining the quality of building blocks. Building blocks fired at 1050°C with up to 20% EMHS in soil were usable for non-loading applications such as ornamental bricks, decorative purpose and fence of garden.

Key Words: EDXRF, TG/DTA, SEM, Shrinkage, Water adsorption, Compressive strength

### 1. Introduction

The degradation of the environment due to discharge of polluting sludge from textile industries is a real problem in several countries and this situation is even worse in developing countries like

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Bangladesh where little or no treatment is carried out before disposals. Moreover as various steps are taken to maintain and improve the quality of surface waters by government bodies, the quantities of sludge generated by these industries continue to increase. Thus industries are confronted with an urgent need to develop safe and feasible alternative practices for sludge management. The overall characteristics of textile sludge depend on the source of the wastewater, industrial types, and wastewater treatment facilities. It contain a wide range of component including organic and inorganic matter, bacteria and viruses, oil and grease, nutrients such as nitrogen and phosphorus, heavy metals and compounds (Thomson et al., 1999). Each component of the sludge has its own impact towards environmental and health of the people living within the immediate vicinity of the sludge disposal location (Thomson et al., 1999).

Electrocoagulated metal hydroxide sludge (EMHS) generates during treatment of polluted effluent by electrocoagulation (EC) technique (Golder et al., 2006). In EC sacrificial anodes (aluminum or iron electrodes) corrode to release active coagulant precursors. Coagulant produces insoluble metallic hydroxide flocs which can remove pollutants by surface complexation or electrostatic attraction (Mollah et al., 2001). The coagulants are in the form of both monomeric hydroxide ions and highly charged polymeric metal hydroxyl species, e.g.,  $\text{Fe}(\text{H}_2\text{O})_6^{3+}$ ,  $\text{Fe}(\text{H}_2\text{O})_5(\text{OH})^{2+}$ ,  $\text{Fe}(\text{H}_2\text{O})_4(\text{OH})_2^{+}$ ,  $\text{Fe}_2(\text{H}_2\text{O})_8(\text{OH})_{24}^{+}$  and  $\text{Fe}_2(\text{H}_2\text{O})_6(\text{OH})_{44}^{+}$ , for anodes made of iron (Amirtharajah and Mills, 1982). These species neutralize the electrostatic charges on the suspended solids and facilitate agglomeration resulting in separation from the aqueous phase by producing EMHS.

In our country all types of sludge are disposed in landfill site haphazardly or openly which leads to soil, surface water and groundwater contamination. Therefore, development of new technologies to recycle, reuse and convert waste materials into reusable materials is critically important for protection of our environment and sustainable development of our society. While landfills are commonly used for disposal of sludge, rapid urbanization has made it increasingly difficult to find suitable landfill sites in Bangladesh and incineration is costly. At places, it is disposed off to nearby rivers or low laying areas, which is likely to pollute surface and groundwater. A possible long-term solution appears to be recycling of the EMHS sustainably and using it for beneficial purposes. One technique that is available is solidification that stabilizes and solidifies components of waste. The solidified product is disposed off to a secure landfill site or it can be recycled and reused as construction materials like bricks, concrete or building blocks if it meets the specific strength requirement (Rahmat, 2001). Utilization or reuse of EMHS as construction and building materials or building blocks is a win-win strategy because it not only converts the waste materials into useful materials but also alleviates the disposal problem. The prospective benefits of using sludge as building blocks additives include immobilizing toxic and heavy metal in the fired matrix, oxidizing organic matter and destroying any pathogen during the firing process and reducing the frost damage based on the results of several full or bench scale studies (Tay and Show, 1999; Lin and Weng, 2001; Weng et al., 2003). Although there are works on sludge of sewage, paper industry, common effluent treatment plant of textile industry, electroplating industry, oil and petroleum industry to make constructional materials i.e., brick, blocks, roofing materials, concretes, tiles (Alleman et al., 1990; Trauner, 1993; Tay and Show, 1999; Lin and Weng, 2001; Weng et al., 2003; Liew et al., 2004; Balasubramanian et al., 2006; Chen and Lin, 2009; Ha et al., 2008; Ismail et al., 2010), but there is no work on EMHS to reuse

it into such materials. Present study systematically explores the reuse potentiality of EMHS generated from effluent treatment by EC in a composite textile industry as an alternative material applied as partial replacement of soil in manufacturing of building blocks. . In order to get quality products, the influence of sludge proportion in building blocks and firing temperature were studied. Prior such investigation geo engineering, elemental, thermal and morphological properties of EMHS were also analyzed.

## **2. Materials and Methods**

### ***2.1. Samples collection and preparation***

The wet EMHS samples were collected from Adury Knit Composite Ltd., (Geographic Location: 24<sup>0</sup>02' N latitude and 90<sup>0</sup>44' E longitude), Narshingdi, Bangladesh. The samples were stored into separate plastic container and kept at ambient temperature prior to analysis. Soil sample was collected from local brick field. For geo-engineering properties investigation, EMHS was mixed with soil from 10% to 40% on wet weight basis. Pure sample that contained 100% soil and 100% EMHS, was also used for comparison. After sun dried of EMHS and EMHS amended soil, samples were made powder using a grinder. Samples that passed through a 20 mesh sieve were taken for elemental, thermal and microstructure analysis.

### ***2.2. Analysis of different properties***

British Standard 1377 (1990) was applied for determination of geo-engineering properties. For this regard moisture content, specific gravity, liquid limit, plastic limit and linear shrinkage were determined by oven-drying method, small pyknometer method, fall cone penetrometer, rolling thread method, respectively. ARL QUANT'X EDXRF (Thermo Scientific, USA) that was equipped with a Rhodium (Rh) anode along with an assembly of eight filters (Al, Cellulose, Cu thick, Cu thin, No, Pd medium, Pd thick and Pd thin) and a Si (Li) detector (with a 15 mm<sup>2</sup> area and ≤76 micron Beryllium window) was used for the elemental analysis of EMHS, soil and EMHS amended soil. Thermogravimetric or thermal profile of the samples was analyzed by a Thermogravimetric/ Differential Thermal Analyzer, TG/DTA 6300, (Seiko Instruments Inc, Japan) by heating from 30°C to 1150°C in pure nitrogen gas medium under the heating rate of 20°C/min. Surface morphology and microstructure of EMHS were analyzed by Scanning Electron Microscope (HITACHI S-3400N, Japan).

### ***2.3. Preparation and quality assessment of building blocks***

Both the soil and EMHS sample were sun-dried and made powder using a grinder and sieve though 100 mesh. In molding process, these EMHS was added to soil by 10% to 50% in weight basis. EMHS free mixture was also made as reference. Dry mixing was done first and then 10% aqueous solution of sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) was added to make homogeneous paste and to bind the materials in the mixture well. Mixtures were then introduced into a series of building block molds with size of 2"×2"×2". Prod with a wooden rod 32 times in about 16 seconds was conducted to ensure elimination of entrained air. After 24 hours maturation followed by drying at room temperature, different batch of the molded blocks were fired in an electric furnace

(Nabertherm, Germany) at the temperature 950°C, 1000°C and 1050°C for 6 hours maintaining a soaking period of 15 min to get the finished products. Quality of building blocks was assessed in terms of weight loss on ignition, shrinkage on ignition, water adsorption and compressive strength as formula given in Table 1.

Table 1 Name, specific methods and using formula for assessing quality of building blocks

Parameter	Using Formula
Weight Loss on Ignition	$(W_a - W_b)/W_a$ Where, $W_a$ =Weight of building block specimen before firing (gm), $W_b$ =Weight of building block specimen after firing (gm)
Shrinkage on Ignition	$(V_a - V_b)/V_a$ Where, $V_a$ = Volume of building block specimen before firing (cm <sup>3</sup> ), $V_b$ =Volume of building block specimen after firing (cm <sup>3</sup> )
Water Adsorption	$(W_a - W_s)/W_a$ Where, $W_a$ = Weight of building block specimen before water submersion (gm), $W_s$ = Weight of building block specimen after water submersion (gm)
Compressive Strength	Using a hydraulic press (Fred S. Carver Inc, U.S.A).

### 3. Results and Discussion

#### 3.1. Different properties of EMHS

Basic geo-engineering properties of EMHS and EMHS amended soil is given in Table 3. As more EMHS was added in the soil, these properties showed a general increasing trend. Specific gravity of soil and clay in their natural state depends on their mineral composition, particle size, distribution of components, texture, resulting void ratio and moisture contents. In addition of EMHS, specific gravity of the mixture was increased due to presence of very high iron content in EMHS.

Average Fe content in EMHS was 923.81 g/kg and this exceeds the allowable range to dispose in agricultural land. Exposure of iron from the sludge into soils may contaminate it and change the soil structure and thus make it harmful for cultivation (Islam et al., 2009). Present study found very high Fe content in EMHS because in EC treatment system iron electrodes were used and these electrodes dissociated and removed pollutants from wastewater and hence produced sludge that contained all iron content of electrodes as well as from various chemicals used in the industry. Hematite, rust, magnetite, lepidocrocite, ferroxihite, maghemite and goethite have been identified as EC by-products by Parga et al., (2005) and Gomes et al., (2007). High range of Mn content in EMHS makes it unusable in agricultural land (Islam et al., 2009).

Ti, Cu, Ni, and Zn are heavy metal and toxic to environmental health and their average concentration in EMHS was 0.56, 0.53, 0.184 and 0.32 g/kg, respectively. Cr, V, Hg and Sr are also heavy metal and mean concentration varied as 130, 14.5, 166.5 and 66.5 mg/kg, respectively. Cu, Ni, Zn and Cr exceeded the recommended value in case of industrial waste (Awahthi, 2000;

Table 2 Basic geo-engineering properties of EMHS and EMHS amended soil

Addition of EMHS in Soil	Properties						
	Moisture Content (%)	Specific Gravity	Liquid Limit (%)	Plastic Limit (%)	Plasticity Index	Liquidity Index	Linear Shrinkage (%)
0%	43.4	2.7	45	21.3	24.4	0.89	9.92
10%	55.2	2.75	55.5	29.9	25.85	0.86	11.4
20%	70.3	2.77	66.6	33.	35.4	1.04	12.85
30%	73.9	2.8	78	34.3	44	0.9	14.28
40%	77.6	2.82	95	42.1	53.4	0.66	18.31
100%	92.85	2.92	339	62	278	0.13	30.49

CCME, 2003) as well as to apply in agricultural land. Some other heavy metals that were found in EMHS included Nb, Cd, and Ga and their content in EMHS ranged from 20 to 35 mg/kg, 11 to 18 mg/kg and 0 to 24 mg/kg, respectively. Cd content in EMHS was higher than the recommended value to apply in the agricultural land (Awahthi, 2000). Cl, Br and S content in EMHS ranged from 1.03 to 3.2 g/kg, 0 to 18 mg/kg and 10.6 to 14.9 g/kg, respectively. So in overall, pre-treatment process for reducing the amount of heavy metal is essential before the EMHS can be used as a soil conditioner in the agricultural land. Soil sample contained 603.6 mg/kg Si while Fe was 104.05 g/kg.

Figure 1 illustrated the TG, DTA and DTG curves of EMHS and soil, respectively. The nature of TG trace showed the 0.5% initial loss of weight from room temperature to 45°C due to removal of moisture content (Wang and Xue, 2010). The onset of temperature 23.3% weight loss occurred from 45°C to 650°C due to dehydration and volatilization (removal of volatiles) of the sample. From 650°C to 901°C, about 5.5% weight loss occurred (Wang and Xue, 2010). Up to programmed temperature, 29.3% weight of sample was degraded and 70.7% weight remained. DTA thermogram showed two endothermic peaks at 90°C and 382°C due to moisture and thermal degradation of sample, respectively. DTG curve showed three distinct peaks at 87°C, 308°C and 811°C. The initial peak was due to mass loss of absorbed moisture at the rate of 0.112 mg/min. The second peak appeared which is attributed to thermal degradation of volatiles materials presented in the sample at the rate of 0.066 mg/min. The third peak arrived due to some oxidation of sample at 0.051 mg/min. It was indicated that EMHS was thermally stable and in dry weight basis 29.3% weight loss occurred at temperature up to 1100°C and this may particularly due to presence of high Fe content. TG curve of soil showed only 2.9% weight loss up to programmed temperature and 97.1% weight remained. DTA thermogram showed two endothermic peaks at 86°C and 457°C due to moisture and thermal degradation of sample, respectively. In DTG curve, only one distinct peak was found at 78°C due to loss of adsorbed water of soil surface at a rate 0.026 mg/min.

Figure 2 showed the SEM micrograph of EMHS at an accelerating and deceleration voltage 15000 volt and 0 volt, respectively with a working distance 10000 µm and emission current 61000 to 66000 nA. EMHS sample was amorphous in nature with edge and porous in body surface. Micrometer sized particles that were generated during treatment of dye were mostly

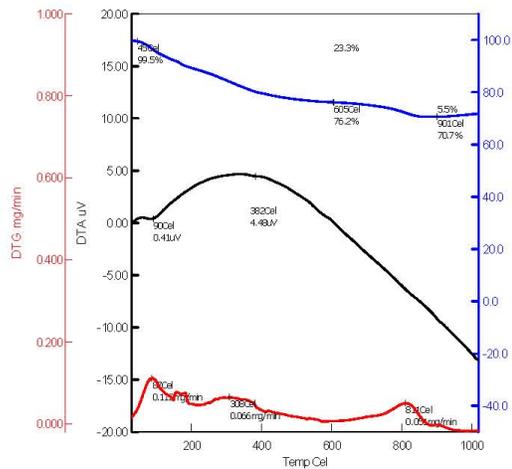


Figure 1 TG/DTA profile of EMHS

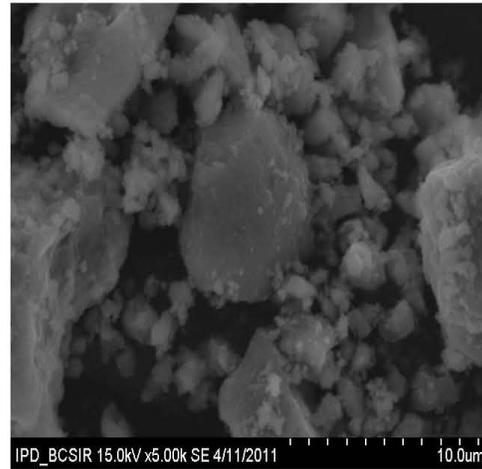


Figure 2 SEM micrograph of EMHS

amorphous in nature (Mollah et al., 2010). Maximum particles were irregular in shape with sharp and hard surfaces. Some pores on the surface were observed, filled with a number of small grains and forming a complex structure. Due to this surface structure, they may have resistance to weathering.

### 3.2. Quality of building blocks

A good building block should be hard. Initially, building blocks were considered unsuitable if building blocks developed cracks while drying; if building blocks developed cracks after firing at any of the temperatures tested; or if deformation in the shape or size of bricks was observed (Roy et al., 2007). All the building blocks of the present work were in good shape and size and no deformation was observed at any firing temperature. After firing at different temperatures, weight loss on ignition, shrinkage on ignition, water adsorption and compressive strength were the main criteria for assessing the overall quality of building blocks.

#### 3.2.1. Weight loss on ignition

Figure 3(a) showed the effect of EMHS addition on the weight loss on ignition of normal building blocks after firing at different temperatures. As more EMHS was mixed with soil and fired at higher temperatures, more weight loss occurred. Weight loss generally occurred due to the evaporation of water from products, melting of inorganic substances and combustion of organic matter during the firing process (Lin and Weng, 2001; Weng et al., 2003; Roy et al., 2007). A first-class brick has a maximum weight loss on ignition of 15%. So from the present study, it was indicated that normal building blocks contained up to 30%, 20% and 10% EMHS and fired at 950°C, 1000°C and 1050°C, respectively, fulfill the standard criteria. Esthetically, the surface texture of the building block was moderately even with the appearance of low small pores, thereby it can be used as facing bricks as well as ornamental bricks (Mahzuz et al., 2009).

### 3.2.2. Shrinkage on ignition

The quality of building blocks can be also assured according to the degree of firing shrinkage. Normally a good quality brick exhibits shrinkage below 8%. Because the swelling of the clay is much lower than that of sludge, an addition of sludge to the mixture widens the degree of firing shrinkage as result the quality of products is downgraded (Weng et al., 2003). Normal building blocks produced from soil had shrinkage of 8.1%, 8.5% and 12.9% at firing temperature of 950°C, 1000°C and 1050°C, respectively. As firing temperature and portion of EMHS in soil increased, the shrinkage also increased as shown in Figure 3(b). The brick shrinkage on ignition is not only attributed to the organic matter content in the clay, but it also depends on the inorganic substance in both clay and sludge being burnt off during the firing process (Lin and Weng, 2001; Weng et al., 2003).

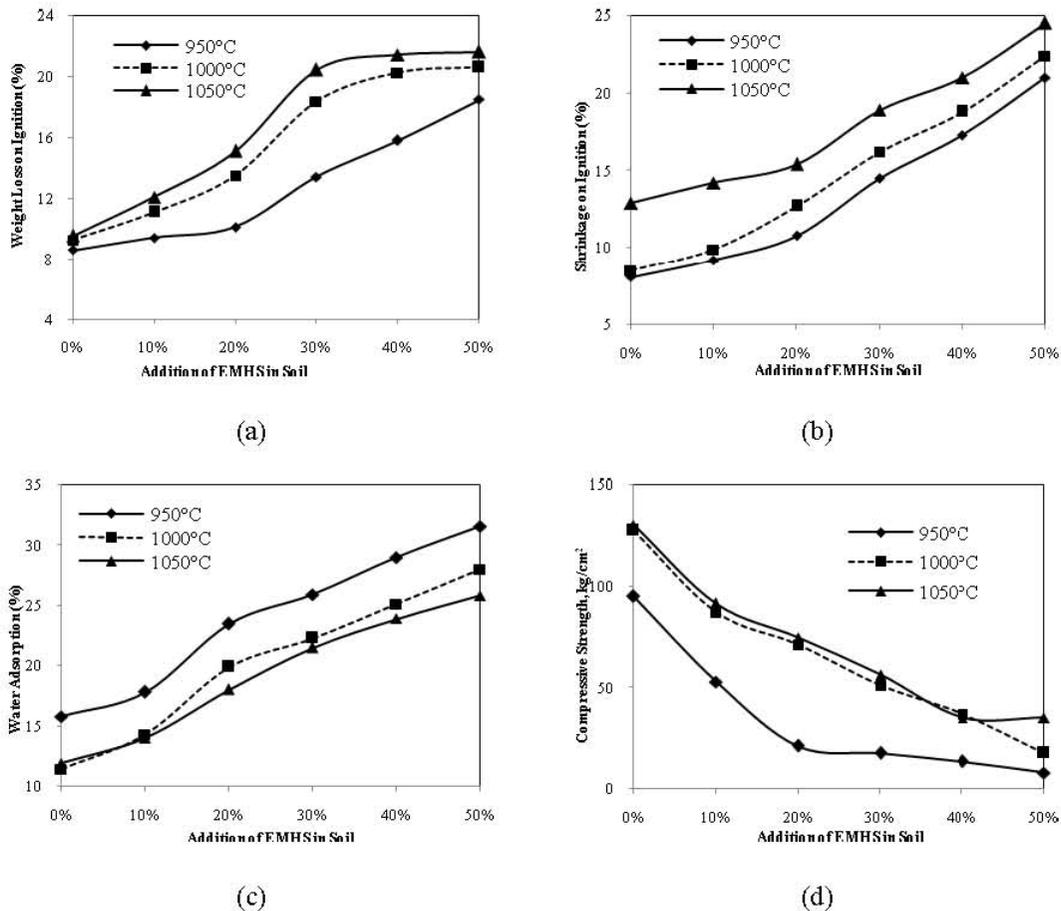


Figure 3 Quality of building blocks- (a) weight loss on ignition, (b) shrinkage on ignition, (c) water adsorption, (d) compressive strength at different EMHS percentage and firing temperature

### 3.2.3. Water adsorption

Water adsorption is a key factor affecting the durability of various types of building blocks and bricks. The degrees of firmness and compaction of building blocks, as measured by their water adsorption characteristics, vary considerably depending on factors such as the type of clay and methods of production used. Low water infiltration into the brick indicates good durability of the brick and resistance to the natural surroundings. Figure 3(c) showed the results of the water adsorption tests of building blocks. The value of water adsorption is proportional to the quantity of EMHS added. Increasing the firing temperature resulted in a decrease of water absorption, thereby increasing the weathering resistance. According to the criterion of water adsorption of bricks in ASTM (1998), the ratio is below 17% for first-class brick and 17 to 22% for second-class brick. According to this guideline building block with 10% EMHS burnt at 1000°C to 1050°C were in first class category. Normal building block with 10% EMHS fired at 950°C, with 20% EMHS fired at 1000°C and with 20% and 30% EMHS fired at 1050°C fall within second-class category. The building blocks exhibited general compliance with some criteria for load bearing bricks under class 1 and 2 because there are no specific requirements in these classes (Liew et al., 2004).

### 3.2.4. Compressive strength

The strength of a material, in general terms, is its ability to resist forces at failure. The strength of dry building blocks was obviously higher than that of wet state. The results of the compressive strength test of the building blocks made from both soil and EMHS mixtures and fired at different temperature was shown in Figure 3(d). The results indicated that the strength was greatly dependent on the amount of EMHS in the building blocks and the firing temperature. Compressive strength of building blocks decreased with increase of EMHS in the mixture but increased with the increase of firing temperature.

As EMHS is not suitable for agricultural land application, it can be utilized in other beneficial purposes. One technique is solidification that stabilizes and solidifies components of waste sludge. The solidified product is disposed off to a secure landfill site or it can be recycled and reused as construction materials like bricks, concrete or building blocks if it meets the specific qualities. Although the blocks did not comply with 1<sup>st</sup> or 2<sup>nd</sup> class standard, normal building blocks with 20% EMHS can be prescribed for the uses of non-loading purposes such as ornamental bricks, decorative purpose and fence of garden etc..

## 4. Conclusion

As the amount of sludge produced by wastewater or effluent treatment plant increases, effective reuse and safe disposal of sludge becomes an important issue because accumulation of sludge is not only a burden to the industry but also affects the environment adversely. Potential reuse feasibility of EMHS in manufacturing of building blocks as a partial replacement of soil were investigated in present study. To assess the quality of blocks weight loss on ignition, firing shrinkage, water adsorption and compressive strength of the manufactured blocks were investigated. EMHS proportion and firing temperature were the two key factors determining the quality of building blocks. At 1050°C firing temperature, up to 20% and 30% EMHS can be applied

in soil for normal building blocks, respectively to manufacture good quality blocks and these blocks can be easily used of non-loading purposes such as ornamental bricks, decorative purpose and fence of garden.

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