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Characterization and Utilization of ETP Sludge of an Automobile Industry for Development of Construction Product

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Abstract: *Laboratory scale studies were carried out for partial utilization of ETP sludge generated from an automobile industry for the development of building products. The chemical properties were determined using XRF show that it is organic and hazardous in nature as it contain phosphate apart from heavy metal ions. Various percentage of ETP sludge (8-15 %) have been used with cement and aggregates for development of road paving blocks by vibro-compaction method. The testing of paving blocks have been carried out for compressive strength and water absorption. It is observed that mixes having 10-11 % of ETP sludge and 22.5-25.0 % of cement give compressive strength of more than 20 MPa and water absorption less than 6 % and were recommended for paving blocks manufacturing. The results of TCLP studies for recommended mixes show that leaching of heavy metal ions (Cr and Ni) in acidic medium are under the permissible limit of USEPA.*
Keywords: *recycled coarse aggregates, paving blocks, compressive strength, flexural strength, C & D waste, natural aggregates*

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I. Introduction

Development of any country leads to rapid industrialization resulting in the problem of environmental degradation. Although, with the advent of pollution control technologies, the industries are able to combat air and water pollution, but the treatment of industrial effluents invariably results in the generation of large volume of the sludge transferring pollutants from liquid phase to solid phase. The indiscriminate disposal of the sludge from effluent treatment plants (ETP) deteriorate fertile surface soil as well as contaminate ground and surface water, which become an important environmental and public health issue. Besides collection, transport and storage of wastes, construction of secured land fill sites pose problems of land acquisition, high land and construction cost, closure of site, environmental monitoring etc. Therefore, it is now a global concern to find a socio, techno-economic and eco-friendly solution to dispose industrial solid wastes.

With the increase in population and urbanization, the demand of raw materials such as cement and aggregates has increased many folds in the construction industry all over the world. The high use of these raw materials has created a significant impact on environment and society [1]. The mission of sustainable development has led to a pressure demand for improving environmental performance in the construction process by reducing consumption of natural resources extracted. The recycling of industrial solid wastes as substitute for building materials is not only environment friendly but also cost effective alternative way to sustain a cleaner and greener environment. Considering the environmental concern, the use of industrial solid wastes, especially, use of ETP sludge as a partial supplement to building materials plays an important role and it is gaining a great momentum.

The cementitious systems based on ETP sludge obtained from various sources have been investigated by various researchers [2-3]. Chin-Haung Weng et al [4] manufactured bricks from dried sludge and observed that good quality bricks can be manufactured using 10 % sludge with 24 % moisture content and fired at 880-960^{o}C. Balasubramanian et al. [5] have suggested that the use of textile ETP sludge up to a maximum substitution of 30% for cement may be possible in the manufacture of non-structural building materials. Singhal et al. [6] replaced cement with lime treated spent pickling liquor sludge (7.5 %) and fly ash (15 %) for development of M 20 concrete. It was observed that the concrete gave optimum compressive strength and comply with the toxicity limits. Shivanath et al [7] carried out a systematic study to utilize effluent treatment plant (ETP) sludge of an automobile, engineering and lead battery industry as partial replacement for cement in M20 concrete. The cement in the concrete mix was replaced by 5, 10 and 15 weight percentage of ETP sludges. At optimum sludge content of 5 %, the compressive strength of concrete were 47.6, 52.9 and 49.8 N/mm^2 for automobile industry engineering and lead battery ETP sludge respectively as against 50.4 N/mm^2 for the control without sludge.

Garg et al [8] conducted experimental studies for characterization and utilization of automobile industry ETP sludge on the properties of cement–sludge binder, paver blocks and flooring tiles. These results

showed that 35 % of automobile sludge be utilized for replacement of fine aggregate in the development of building components.
The results of leaching studies, conducted for tiles/paver blocks samples revealed that the concentration of leached metals is quite low than the limits specified USEPA.

In the automobile industry, the effluent is mainly generated from degreasing, phosphating and painting operations and in India, about 3.0 million tonnes per annum ETP sludge is generated from automobile industries [8] and disposed on land. This enormous amount of disposed sludge contain impurities of inorganic salts and toxic metals which is a threat to environment. Therefore, in the there is a need to carry out a systematic study to utilize automobile industry sludge as construction to disposal and reduce pollution. In the present investigation, study has been carried out to utilize automobile effluent treatment plant (ETP) sludge as replacement of fine aggregate for development of road paving blocks and determination of engineering properties as per Indian Standards. Toxicity characteristics leaching procedure studies have also been carried out as per USEPA to determine the leaching behaviour of hazardous metals from the blocks for application purpose.

II. Materials And Methods

2.1. Cement

Ordinary Portland Cement (OPC) of 43 Grade conforming to IS: 8112:1989 [9] was utilized as binder material for development of concrete paving blocks and typical physic-chemical composition of OPC has been shown in Table 1.

2.2. Coarse and fine aggregates

The locally available natural crushed stone, generally of quartzite type has been used as natural coarse aggregate (NCA) of maximum 10 mm size satisfying the grading requirements of IS 383: 1970 [10]. The particle size distribution along with physical and mechanical properties of natural aggregates determined as per IS 2386:1963 [11] is given in Table 2. Local river sand complying with the particle size requirements of IS 383: 1970 were used as natural fine aggregates (NFA) for fabrication of blocks. The physical properties of fine aggregates have been given in Table 3.

2.3 ETP Sludge Collection and Characterization

The sample of ETP sludge was collected from a Pune based automobile industry in which the industrial effluent and the domestic effluent is treated (secondary treatment) collectively after primary treatment. The sample was dried at 50 ± 2^{o}C in trays, cooled to room temperature, ground in a ball mill to a fineness of passing 300 µm sieve. The particle size analysis of ETP sludge is shown in Table 4. The sample was analyzed for various physical and chemical properties and the results are shown in Table 5 and 6 respectively.

Table 1: Physico-chemical Analysis of OPC (43 Grade)

Parameters	Values
Physical Properties	
Loss on ignition	3.60 %
Consistency	31.25 %
Soundness	1.0 mm
Bulk density	1.4 g/cm^3
Initial setting time	172 min
Final setting time	308 min
Specific Gravity	3.14
Compressive strength (28 day)	49.5 MPa
Chemical Properties	
SiO_2	21.45 %
Al_2O_3	5.25 %
Fe_2O_3	3.70 %
CaO	61.45 %
MgO	2.85%
SO_3	3.00 %
Insoluble residue	4.40%

Table 2: Physical and mechanical properties of coarse aggregate

Sr. No.	Properties	Value
1.	Flakiness index	9.1 %
2.	Elongation index	12.5 %
3.	Water absorption	0.40 %
4.	Specific gravity	2.7 %
5.	Bulk density	1.560 g/cm^3
6.	Crushing value	13.2 %
7.	Impact value	9.5 %
8.	Particle size distribution	
	Size	**Passing (%)**
	20 mm	100
	12.5 mm	92
	10 mm	22.4
	4.75 mm	1.4
	2.36 mm	0.1
	1.18 mm	0.1
	600 µm	0.1
	300 µm	0.1
	150 µm	0.1
9.	Fineness modulus	6.84

Table 3: Physical properties of fine aggregates

Sr. No.	Properties	Value
1.	Specific gravity	2.7
2.	Water absorption	4.50 %
3.	Bulk Density	1.5 g/cm3
4.	**Particle size distribution**	
	Size	**Passing (%)**
	10 mm	100
	4.75 mm	99.75
	2.36 mm	76.65
	1.18 mm	54.35
	600 µm	44.5
	300 µm	33.0
	150 µm	16.55
5.	Fineness modulus	2.7

Table 4: Particle size analysis of ETP sludge

Particle size (µm)	Percentage (%)
> 600	Nil
300-600	23.9
300-150	58.6
75-150	13.2
45-75	4.3

Table 5: Physical characteristics of ETP sludge

Sr. No.	Parameters	Value
	Physical parameters	
1	pH	7.82
2.	Colour	Light grey
3.	Bulk density	0.76 g/cm^3
4.	Specific gravity	0.69
5.	Ash content (as dry basis)	62.9 %
6.	Gross calorific value (dry basis)	1370 kcal/kg
7.	Moisture content	10.1 %
8.	Volatile solids	2.3 %
9.	Fixed solids	5.3 %
10.	LOI at 550^{o}C	37.1 %

Table 6: Chemical analysis of ETP sludge

Sr. No.	Parameters	Value
1.	SiO_2	7.4 %
2.	Al_2O_3	6.7 %
3.	Fe_2O_3	7.2 %
4.	CaO	48.3 %
5.	MgO	1.6 %
6.	ZnO	1.5 %
7.	MnO	0.8 %

8.	Cr_2O_3	0.03 %
9.	P_2O_5	19.5 %
10.	Ni_2O_3	0.7 %
11.	SO3	3.5 %
12.	Cl^-	1.1 %
13.	K_2O	0.5 %
14.	PbO	0.06 %
15.	CuO	0.05 %
16.	Carbon as C (as dry basis)	18.8 %
17.	Hydrogen as H (as dry basis)	3.7 %
18.	Nitrogen as N (as dry basis)	1.3 %
19.	Sulphur as (as dry basis)	0.4 %
20.	Total organic carbon (TOC)	21.1 %

2.4 Mix Composition of Road Paving Blocks

The fabrication of paving blocks was carried out using compaction method following the procedure and specifications described in IS: 15658: 2006 [12]. Precast concrete blocks for paving-specifications. Since zero slump concrete is used in production of paver blocks, the quality of blocks will depend upon various parameters like the capacity of compaction and vibration of machine, grade of cement used, water content, quality of aggregates used, their gradation and mix design adopted, handling equipment employed, curing methods adopted, level of supervision, workmanship and quality control achieved etc.

Two layered paving blocks were fabricated using cement, coarse aggregates, stone dust as fine aggregates and water. The top layer was prepared using cement and stone dust in a proportion of 2:1 and water as added to 9 %. The composition of top layer remains same in all the mixes. For fabrication of bottom layer, various concrete mixtures were tried using cement (OPC 43 Grade) as binder varying from 20-25 %, ETP sludge as fine aggregate (stone dust) ranging from 10-15 % and coarse aggregate ranging from 37.5 to 50 %. Water content added was varied from 11.50 to 12.0 % and all the trial mixtures were expected to achieve a compressive strength of 10-15 MPa after 28 days of curing. The mix compositions used and their designations for control and ETP sludge replaced mixes are given in Tables 7.

Table 7: Mix proportion of paving blocks

Mix designations	Cement (%)	Stone dust (%)	ETP sludge (%)	Coarse aggregate (%)	Water (%)
Top Layer	33.33	66.67	----	----	9.00
A0 (Control)	**25.00**	**37.50**	**----**	**37.50**	**8.00**
A1	25.00	27.50	10.00	37.50	12.00
A2	25.00	26.50	11.00	37.50	12.00
A3	25.00	25.50	12.00	37.50	12.00
A4	25.00	22.50	15.00	37.50	12.00
B0 (Control)	**22.50**	**33.50**	**----**	**44.00**	**7.5**
B1	22.50	23.50	10.00	44.00	12.00
B2	22.50	22.50	11.00	44.00	12.00
B3	22.50	21.50	12.00	44.00	12.00
C0 (Control)	**20.00**	**30.00**	**----**	**50.00**	**7.0**
C1	20.00	20.00	10.00	50.00	11.50
C2	20.00	18.00	12.00	50.00	11.50

2.5 Mixing, Fabrication and Curing of Paving Blocks

The fabrication of paving blocks was carried out using compaction method following the procedure and specifications described in IS: 15658: 2006 [12]. For fabrication of 200 x 120 x 80 mm block, the desired quantity of cement and stone dust mixed with water was filled in the mould to achieve the thickness of top layer was kept in the range of 4-6 mm. For bottom layer, the mixing of materials was performed in a drum mixer using conventional method. The weighed quantities of cement and ETP sludge were poured in the drum and allowed to dry mix for one minutes. Subsequently half of the measured quantity of water was added to the bulk materials as per respective mix and the mixture was further mixed for two more minutes for development of cementitious layer around the ETP sludge particles. Now the measured quantities of coarse aggregates and fine aggregates were added in the drum along with remaining half of the water and all these materials were mixed for 2 minutes. After filling the mould, a hydraulic pressure of 50 tonnes was applied for 10-15 seconds on the mixture for compaction. After releasing the pressure, the paving block was removed from the mould and then cured at a relative humidity of over 90 % at room temperature (25±2^{o}C) for 28 days. The cured blocks were tested for physical and mechanical properties as per IS: 15658: 2006.

2.6 Engineering Properties of Blocks

The physical and mechanical properties of paving blocks like water absorption and compressive strength were determined after 28 days curing period of all the mixes and compared with control.

Water absorption is the measure of permeability and porous nature of hardened concrete and is determined after 28 day of curing as per method described in IS: 15658:2006. The blocks of all mixes were completely immersed in water at room temperature for 24 h. After removal from water, blocks were allowed to drain for 1 min by placing them on a 10 mm wire mesh, and visible water was removed with a damp cloth and immediately weighed. Subsequent to saturation, the blocks were dried in ventilated oven at 105±2^{o}C for 24 h. The blocks were weighed after cooling at room temperature to calculate the water absorption and the average of three specimens was reported.

Mechanical properties like compressive strength was determined according to the procedure described in IS: 15658: 2006 after 28 days curing and the average values of three specimens tested for each mix were reported in the results. Before determination of compressive strength, all the specimens were stored for 24 h in water at room temperature of 25±2^{o}C, air dried and tested. The corrected compressive strength has been calculated by multiplying the apparent strength with correction factor for thickness and arris/chamfer of paving block according to IS: 15658.

2.7 Methodology of TCLP

The TCLP tests were conducted for 28 and 90 days old hydrated samples (A1, B1 and C1). To conduct the test, a slice of approximately 9.5 mm thick was cut from the mid height of the product. A part of each slice weighing approximately 100 g was crushed carefully using a hammer so that all the particles were <9.5 mm, but the variation on the particle size distribution was not significant. Leaching studies were carried out in water as well as in severe acidic conditions (pH=~2.88). 200 ml of water or acetic acid solution were added to the 10 g of sample (liquid to solid ratio 20: 1) in a high-density polyethylene bottle. The bottle and its contents were agitated in a rotary shaker at 30 rpm for 18 h. The leachates were filtered through a 0.45 μm membrane filter to remove suspended solids and the leached solutions were used for determination Cr, Ni, Mn, Zn and Pb by Inductive Couple Plasma spectrophotometer. Each leachate was analysed in duplicate and average values were reported to ensure the reproducibility of the data.

III. Results And Discussion

3.1. Visual inspection

After 28 days of curing period, visual inspection of blocks was carried out in natural day light prior to the tests for other properties. The photograph of fabricated paving blocks has been shown in Fig. 1 (a-b) Visual inspection revealed that all paving blocks were sound and free of cracks. No other visual defects were observed which may interfere with proper paving of the unit or impair the strength or performance of pavement constructed with the paving blocks. The bottom layer was showing proper bonding with top layer in all the paving blocks.

(a) (b)

Fig. 1 (a-b): Photographs of fabrication of road paving blocks

3.2. Engineering Properties of Blocks

The blocks were weighed after cooling at room temperature to calculate the percentage water absorption and the average of three specimens was reported in Table 8. The results show that only mix A1, A2 and B1 are satisfying the requirements (<6 %) of IS: 15658 for water absorption.

Table 9: Test results of physical and mechanical properties of paving blocks

Mix designations	Water absorption (%) (28days)	Compressive strength (MPa) (28 days)	Remark
A0 (Control)	2.0	62.50	
A1	4.20	26.50	**Recommended**
A2	5.50	21.25	**Recommended**
A3	10.00	16.25	
A4	18.75	8.00	
B0 (Control)	2.80	50.50	
B1	5.40	21.00	**Recommended**
B2	12.75	16.50	
B3	16.55	14.25	
C0 (Control)	3.20	45.00	
C1	15.50	13.25	
C2	20.00	10.00	

The results of compressive strength of various mixes are shown in Table 8. It has also been observed that the compressive strength of all the blocks prepared decreases from 26.50 to 8.0 MPa with increase in sludge content from 10 to 15 %. It is due the presence high organic matter and phosphate content present in the sludge which is very much deleterious for strength development reactions of cement. The recommended mix compositions are A1, A2 and B1 for blocks manufacturing which may be used for light vehicles movement as well as for inter pavement having compressive strength more than 20 MPa.

3.3 TCLP Studies

TCLP leaching tests of final recommended mixes for the paving blocks and bricks utilizing ETP sludge were carried out at 28 days and 90 days of curing. The results of leaching behavior of metal ions in water as well as in acetic acid (pH=~2.88) are shown in Table 9 for paving blocks along with the prescribed limits by U.S. EPA. The results shows most of the hazardous elements are immobilized in cement matrix and the metals coming in leachate (Ni and Cu) are well below the prescribed limits of US EPA.

Table 9: Leaching characteristics of metals from paving blocks

Mix	Metal ions (mg/l)	Water		Acid		U.S. EPA TCLP Standards
		28 days	90 days	28 days	90 days	
A1	Cr	BDL	BDL	0.065	BDL	5.0 mg/l
	Mn	BDL	BDL	BDL	BDL	NA
	Ni	1.95	1.77	4.86	1.89	NA
	Zn	BDL	BDL	BDL	BDL	NA
	Pb	BDL	BDL	BDL	BDL	5.0 mg/l
A2	Cr	BDL	BDL	BDL	BDL	5.0 mg/l
	Mn	BDL	BDL	BDL	BDL	NA
	Ni	2.06	1.97	2.81	2.55	NA
	Zn	BDL	BDL	BDL	BDL	NA
	Pb	BDL	BDL	BDL	BDL	5.0 mg/l
B1	Cr	BDL	BDL	BDL	BDL	5.0 mg/l
	Mn	BDL	BDL	BDL	BDL	NA
	Ni	2.55	2.16	3.44	2.89	NA
	Zn	BDL	BDL	BDL	BDL	NA
	Pb	BDL	BDL	BDL	BDL	5.0 mg/l

IV. Conclusions

The investigation carried out for development of paving blocks using ETP sludge in different compositions revealed following conclusions:

1. The chemical characterization of ETP sludge shows that it contain high phosphate content which is organic in nature and is deleterious for hydraulic reactions of cement.
2. ETP Sludge can be a successful partial replacement material for natural sand in the manufacture of cement paving blocks.
3. Optimum amount of 10-11 % of the ETP sludge can be utilized for block development in using 22.50-25.0 % of cement as a binder material as these mixes have the strength of >20 Mpa and water absorption <6%.
4. The blocks can be manufactured on commercial basis as the leaching of heavy metal ions in TCLP studies (Cr and Ni) are under the limit of USEPA.
5. Thus, the ETP Sludge utilization may be helpful in the reduction of environmental pollution and its disposal problems.

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