Statistical Design of Portland cement Modified with Water Treatment Plant Sludge

Yousif Algamal, N. M. Khalil, Mustafa M. Mohammed

Abstract: ***One-Way Analysis of Variance (ANOVA) was applied in this investigation to study the feasibility of modifying ordinary portland cement through Water treatment plant sludge (WTSP) addition. Cement pastes were obtained from mixtures of water treatment plant sludge (WTPS), calcined sludge and ordinary portland cement (OPC) using the standard water consistency. The variation in bulk density, apparent porosity, chemically combined water and compressive strength of the prepared samples were determined over time. In most cases the addition of sludge results in a decrease in apparent porosity (26.36 - 20.40) %, but an increase in chemically combined water (22.46- 29.35), bulk density (1.78 - 1.845) g/cm^3 and compressive strength (21- 85) MPa with time. The results show a recognized improvement in densification parameters when using calcined sludge which recommend their use in cement manufacturing so one could expect better economical and environmental benefits.***

The data obtained were analyzed using a one-way (Analysis of Variance) ANOVA. The determinations were performed in triplicate (n= 3), mean ± standard deviation (SD) values were calculated. A comparison between the means of three different groups and four curing time was performed at 5% level of significance. The ANOVA results show that there are statistically significant difference in chemically combined water property between different combination groups whereas insignificant between the different curing times, for bulk density it shows significant difference between groups in curing times and insignificant difference in combination of groups and mechanical strength shows significant difference between group of curing times and insignificant difference in groups of combination finally for apparent porosity property we didn't find any significant difference between groups for combination of groups and curing times for the hardened pastes.

Keywords: ***Water treatment plant sludge, calcined sludge, physical properties, mechanical properties, environmental impact, ANOVA.***

I. INTRODUCTION

Most of the academic society and industrialist paying much attention to preserve the environment particularly in processes involving utilization of pollutant solid wastes. Sludge is a waste or by-product produced from water treatment plants that generated during process of purification of drinking water using different methods (coagulation, flocculation and filtration) [1-4] from raw water, huge amount of sludge generated every year as the need of pure water required by increased population over the world.

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The by-product accompanied the process of purification of raw water or waste in the form of sludge presents several risks for the environment and human health [5,6] due to its difficult biodegradation.

Several methods of treatment were made to consolidate, stabilize and dewater the sludge, in most cases it will be disposed to the nearest watercourse, soil application or buried by landfill around the treatment plant, landfill has become dominant manner of sewage sludge waste [7,8]. However, landfill is only a temporary solution for the disposal of sewage sludge waste because there is limited space for the sludge waste to be disposed.

Recently, several researches were done in this field. Naamane et al [9] studied the characterization of calcined sewage sludge for its incorporation in cement they have shown that the calcined sludges contain different oxides of different sizes, the compositions of calcined sludges recommend their use in cement manufacturing. Tay & Show [10] investigated the use of water treatment sludge and clay for bricks production as construction material. Monzo et al. [11] studied usage sewage sludge ash in mortars. Alleman & Berman [12] used sludge ash in manufacturing of bricks. Bhatty & Reid [13] reported as a fine aggregate in mortars. These studies focus on studying the hydration behavior of ordinary portland cement modified with sewage sludge ash (SSA) and water purification sludge ash (WPSA) [14 - 19].

Several pozzolanic materials such as rice husk ash, fumed silica, fly ash and blast furnace slag are usually used as additives to portland cement to support building materials industry [20, 21]. Nagy & Yousif [22] reported the addition of natural minerals to OPC to increase its productivity. Other alternatives involving agricultural wastes such as periwinkle shell, palm-kernel, corn-cob ash, as well as saw dust ash are also used as cement additives [23-29].

The aim of this paper is to carry statistical evaluation to the possibility of increasing cement production capacity using WTSP as additives.

II. MATERIALS AND METHODS

A. Materials

OPC and WTPS were used as additives to OPC, some general use chemicals e.g. diethyl ether and methyl alcohol were used for hydration stopping at different ages.

a) Processing of water treatment plant (WTP) sludge

The WTP sludge was collected, dewatered; air dried under sunlight, heated at 110 ^{o}C for 3 hours and pulverized using a ball mill, then sieved through 75-µm sieve, to separate the impurities and large particles present in the sludge.

b) Calcination of water treatment plant (WTP) sludge

The raw water treatment plant (WTP) sludge was calcined through heat treatment at 700°C for 1-hour soaking time using (JSR, JSMF-140T) electrical furnace.

c) Characterization of WTP sludge

The WTP sludge used in this study was collected from Al Mogran Water Treatment Plant at Khartoum, Sudan Republic. The plant located at White Nile River near to the point of connection of the White and Blue Niles. The chemical constitution of the sludge was determined using X-ray fluorescence (XRF) technique. The solid phase composition of the sludge was investigated using XRD diffractometer (Bruker, Germany).

B. Sample preparation

Three cement mixes were prepared; one is the reference (zero % sludge additions, the other two mixes containing 30 wt.% of either raw or calcined sludge (Table 1). Three representative samples of the hardened samples from each cement mix were tested for their sintering and mechanical properties according to the International Standard Specification [30], at different ages of hydration up to 90 days.

Table 1. The percentage proportions of cement mixes

Samples	OPC	WTPS	Calcined WTPS
A	100 %	00%	00%
B	70 %	30 %	00 %
C	70 %	00 %	30 %

The chemically combined water was determined and calculated from the following equation:

$$\%\ of\ chemically\ combined\ water = \frac{\mathrm{Wt.\,green} - \mathrm{Wt.\,ignited\ x\ 100}}{\mathrm{Wt.\,green}}$$

C. Instrumentation

X-ray fluorescence (XRF) and X-ray diffraction (XRD) techniques were followed to determine the chemical compositions of the starting materials. XRD was used also to follow the mineral composition of the hydrated cement mixes.

D. Cement pate testing

The suitable amount of mixing water, setting time, sintering parameters (bulk density and apparent porosity) as well as mechanical strength were tested according to the International Standard Specifications [31-34].

III. RESULTS AND DICUSSION

A. Raw materials characterization

a) Chemical constitution of OPC and WTPS

The XRF analysis of the starting materials (Table 2) indicates that the OPC contains 63.50% CaO, 24.50% SiO_2, 7.00% Al_2O_3 and 4.02% Fe_2O_3 with little contents of other constituents. According to the American classification, the cement used in this research work is ordinary Portland cement, Type I [35,36]. WTPS composed mainly of silica (51.22 %), alumina (15.65 %) and ferric oxide (8.14 %) with little contents of other impurities. Its composition is almost similar to brick clay, but with relatively greater iron oxide content, the loss on ignition is 12.95 %, which represent the moisture and organic matter in the raw sample.

Table 2. Chemical compositions of OPC and WTPS

Chemical composition	OPC %	WTPS %
CaO	63.50	2.62
SiO_2	24.50	51.22
Al_2O_3	7.00	15.65
Fe_2O_3	4.02	8.14
MgO	0.08	1.31
Na_2O	0.25	2.86
K_2O	0.25	1.11
SO_3	0.40	--
LOI	3.01	12.95

b) Mineralogical composition of OPC and WTPS

Fig. 1 shows that OPC is composed mainly of calcium silicate (Ca_3SiO_5 - 71.9 %) and calcium aluminum oxide ($Ca_3Al_2O_6$ - 28.1 %). The XRD pattern of the WTP sludge, shown in (Fig. 2) clearly showing the presence of three major crystalline phases of the sludge viz., nontronite [(Na, Ca) O,3Fe_2(Si, Al)$_4$ $O_{10}(OH)_2$. XH_2O], albite [Na (Si_3 Al) O_8] and anorthite [(Ca, Na) (Si, Al)$_4O_8$]. These minerals are mainly silicates and may be useful as additives for OPC.

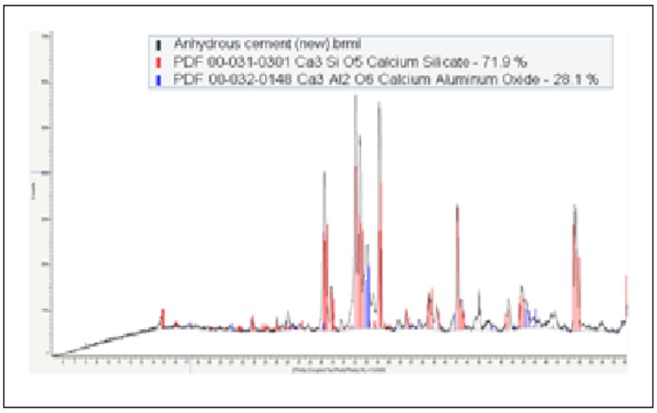

Fig. **1 XRD pattern of ordinary portland cement**

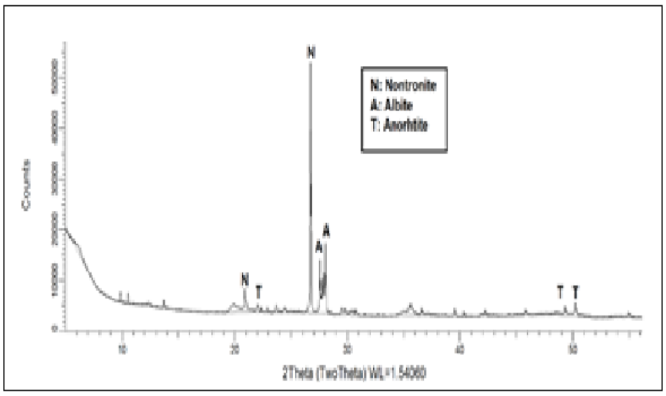

Fig. 2. XRD patterns of the raw sludge

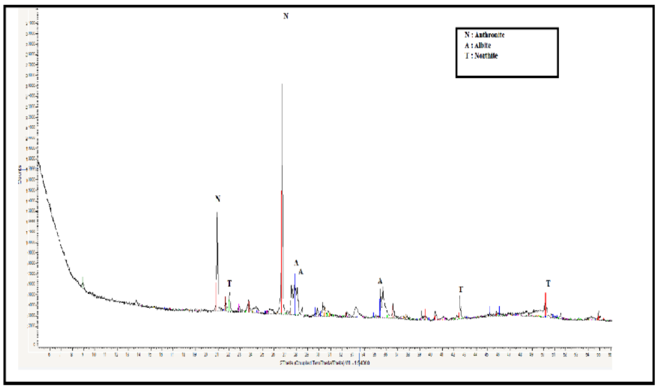

Fig. 3. XRD patterns of the calcined sludge

B. Technological Tests

a) Consistence of gauging water

The required amount of mixing water to produce the cement paste is very sensitive as slight decrease in water content leads to poor consideration and workability while increase in water content leads in loosed matrix and hence poor mechanical strength.
Table (3) shows that different cement mixes consumes different contents of water consistency, due to the difference in fineness and compositions of OPC sample (A) compared with the calcined sludge (C) and that of the sample (B) for the dry WTPS.

Table 3. Water consistency of OPC and sludge pastes

Sample No.	Paste mix	Standard water %
A	Reference	28
B	OPC and dry sludge	34
C	OPC and calcined sludge	40

b) Hydration

Fig. 3 shows a gradual increase in the content of combined water as the curing time increases; 3, 7, 28 and 90 days. From the figure it is shown also that cement paste (C) have relatively higher contents of chemically combined water (22.46,- 29.35) as the calcined sludge had a slightly higher temperature compared with the heat content of OPC paste (A) that recorded (14.59,16.31,18.01 and 19.12) and even less content chemically combined water for the paste mixed with dry sludge i.e., green sample (B) shown the values (13.28,15.21,17.20 and 18.42) with respect with the curing time 3,7,28 and 90 days respectively as a result the chemically combined water will be increased. This can be explained in terms of that the calcined sludge contains the minerals such as calcium aluminate (C_3A) and calcium oxide (CaO) in the sample when heated at 700°C. The chemical constituents of the calcined sample increasing the chemical reactions of oxides and hence increasing the heat content of the pastes.

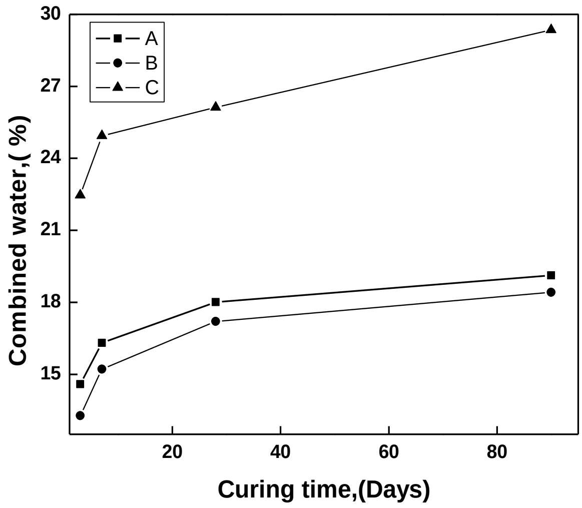

Fig.3, Chemically combined water of the hardened cement

To determine the effect of chemical combined water on different samples, we use One- way analysis of variance, which proved that, there is statistically significant difference between our samples at $p < 0.001$ level for three samples as shown in Table 4, and the values of mean and standard deviation in Table 5 ,[$F(2,9) = 19.8$, $p = 0.001$]. Post- hoc multiple comparison (Table 6), using the Tukey indicated that the mean chemically combined water of hardened cement of samples A and B are significantly lower than sample C. There was no statistically significant difference between sample A and B as shown in the tables 7&8.

Table 4, ANOVA for chemically combined Combined water

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	227.627	2	113.814	19.818	.001
Within Groups	51.687	9	5.743		
Total	279.315	11			

Table 5, Descriptive statistics for chemically combined water

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		

A	4	17.0075	1.98314	.99157	13.8519	20.1631	14.59	19.12
B	4	16.0275	2.25949	1.12974	12.4321	19.6229	13.28	18.42
C	4	25.7175	2.86199	1.43099	21.1634	30.2716	22.46	29.35
Total	12	19.5842	5.03907	1.45465	16.3825	22.7858	13.28	29.35

Table 7, ANOVA for chemically combined

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	49.685	3	16.562	.577	.646
Within Groups	229.629	8	28.704		
Total	279.315	11			

Table 8, Descriptive statistics for chemically combined water

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
3 days	3	16.7767	4.96530	2.86672	4.4422	29.1112	13.28	22.46
7 days	3	18.8200	5.32854	3.07643	5.5832	32.0568	15.21	24.94
28 days	3	20.4433	4.93279	2.84795	8.1896	32.6971	17.20	26.12
90 days	3	22.2967	6.11838	3.53245	7.0978	37.4956	18.42	29.35
Total	12	19.5842	5.03907	1.45465	16.3825	22.7858	13.28	29.35

c) Bulk density of the hardened cement pastes at different curing time

Fig.4 shows that the bulk density increases gradually with increasing the curing time 3,7,28 and 90 days from the results at 90 days it is seen that the densities hardly vary with additions of calcined sludge and have the highest value 1.845 g/cm^3. The values of bulk density of hardened paste (C) recorded the highest values among the samples under the study and recorded (1.758,1.778, 1.812 and 1.845) g/cm^3 with respect to the curing days 3,7,28 and 90 respectively compared with the reference sample (A) values that recorded (1.709,1.761,1.799 and 1,83) g/cm^3 and the lowest values recorded for the green sample (B),i.e.,(1.701 ,1.724 ,1.757 and 1.802) g/cm^3. This can be explained in terms of the compact of the matrix due to the presence of the minerals such as calcium aluminate and calcium oxide in the calcined sludge that bring the closeness of the gaps and filled the holes resulting in solid phase.

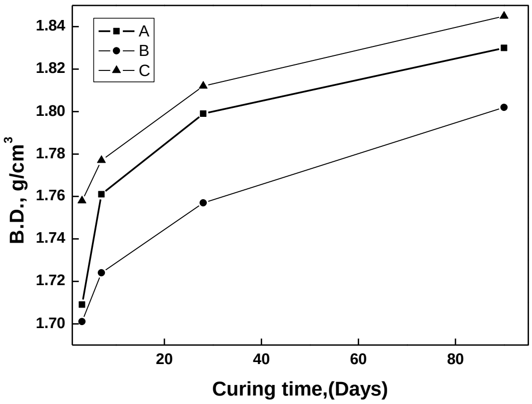

Fig.4, Bulk density of the hardened cement

To investigate the differences between the groups we conducted a One-way analysis of variances to see the impact of days on the bulk density. There was statistically significant difference between groups (Tables 9 & 10) [F (3,8) =7.8, p= 0.009]. Tukey (Table 11) reported that the bulk density in 3days is statistically lower to 90 days, but the test did not show any difference in bulk density between other groups.

Table 9, ANOVA for bulk density

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.018	3	.006	7.851	.009
Within Groups	.006	8	.001		
Total	.024	11			

Table 10, Descriptive statistics for chemically combined water

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean Lower Bound	95% Confidence Interval for Mean Upper Bound	Minimum	Maximum
3 days	3	1.722667	.0308599	.0178170	1.646006	1.799327	1.7010	1.7580
7 days	3	1.754333	.0276104	.0159409	1.685745	1.822921	1.7240	1.7780
28 days	3	1.789333	.0287460	.0165965	1.717924	1.860742	1.7570	1.8120
90 days	3	1.825667	.0218251	.0126007	1.771450	1.879883	1.8020	1.8450
Total	12	1.773000	.0465169	.0134283	1.743445	1.802555	1.7010	1.8450

d) Apparent porosity of hardened cement pastes at different curing time

Fig.5, shows the apparent porosity (The total porosity test determines the durability and long term function of the hardened paste) values of prepared samples of OPC (A), OPC & dry sludge (B), and OPC and calcined sludge (C) the following results showing that apparent porosity decreases gradually with increasing the curing time 3,7,28 and 90 days, the addition of dry sludge increases the porosity and absorption percentage initially decreasing with increasing the curing time as in sample (B), that recorded (26.36,23.56,22.12 and 20.40)%, higher than that of the reference sample (A) the percentage of apparent porosity decreases with increase of curing time, its values (23.54, 22.26, 20.12 and 18.26) %. The values of apparent porosity of hardened paste (C) recorded the lowest values among the samples under the study and recorded (21.24, 19.55, 16.24 and 15.22) % with respect to the curing days 3,7,28 and 90 respectively. This can be explained in terms of that the open porous matrix of the green sample (B) is much loosen compare with that of the reference sample (A) of less pores and still less for the calcined sludge sample (C) which shown the lowest apparent porosity percentages.

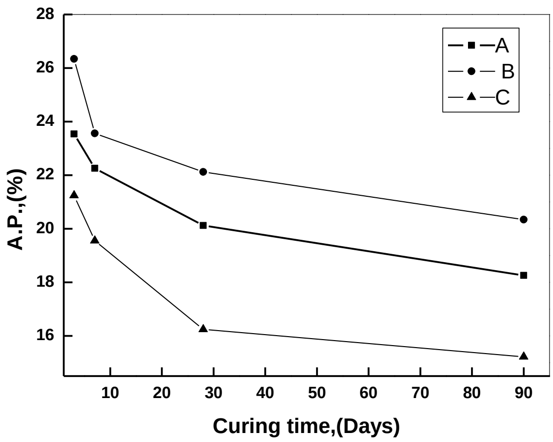

Fig.5, Apparent porosity of the hardened cement

To investigate the differences between the combination of groups as well as combination of curing times we conducted a One-way analysis of variances to see their impact on the apparent porosity. There was no significant difference between groups and the curing times (Tables 12 & 13).

Table 12, ANOVA for apparent porosity

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	51.232	2	25.616	3.879	.061
Within Groups	59.441	9	6.605		
Total	110.672	11			

Table 13, Descriptive statistics for apparent porosity

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean Lower Bound	95% Confidence Interval for Mean Upper Bound	Minimum	Maximum
A	4	21.0450	2.33189	1.16594	17.3344	24.7556	18.26	23.54
B	4	23.0950	2.54408	1.27204	19.0468	27.1432	20.34	26.36
C	4	18.0625	2.81131	1.40566	13.5891	22.5359	15.22	21.24
Total	12	20.7342	3.17193	.91566	18.7188	22.7495	15.22	26.36

To investigate the differences between the groups we conducted a One-way analysis of variances to see the impact of curing days on the apparent porosity. There was no significant difference between groups (Tables 14 & 15).

Table 14, ANOVA for apparent porosity

Apparent porosity

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	58.012	3	19.337	2.938	.099
Within Groups	52.661	8	6.583		
Total	110.672	11			

Table 15, Descriptive statistics for apparent porosity

					95% Confidence Interval for Mean			
	N	Mean	Std. Deviation	Std. Error	Lower Bound	Upper Bound	Minimum	Maximum
3 days	3	23.7133	2.56440	1.48056	17.3430	30.0836	21.24	26.36
7 days	3	21.7900	2.04590	1.18120	16.7077	26.8723	19.55	23.56
28 days	3	19.4933	2.98967	1.72609	12.0666	26.9201	16.24	22.12
90 days	3	17.9400	2.57496	1.48665	11.5435	24.3365	15.22	20.34
Total	12	20.7342	3.17193	.91566	18.7188	22.7495	15.22	26.36

e) Compressive Strength of hardened cement pastes at different curing time

Fig.6 shows the comparison between the compressive strength (that determines the applicability potentials of the paste) of prepared cement samples (without addition, with addition of dry sludge and with the calcined sludge) at 3, 7, 28 and 90 days of curing time to assess the potentiality of WTPS as additive to OPC. The increase in the compressive strength as in the Figure 6 which provides the experimental data and the trend for compressive strength of prepared samples up to 90 days respectively. The hardened sample (C) shown the highest values for mechanical strength (21,35,60 and 85) MPa and the reference sample (A) gave (20,30,45 and 60) MPa and the green sample (B) shown the least mechanical strength values (15,25,40 and 55) MPa when the curing time increases 3,7,28 and 90 days. From these results the compressive strength for the mix calcined at 700^{o}C for 1 h was 85 MPa in 90 days ,which is higher than the values found for the mix prepared from OPC (60 MPa) and that for the dry WTPS cement paste (55 MPa).This can be explained in terms of the presence of minerals in calcined sludge are cementitious or pozzolanic, contribute to improving mechanical strength of the hardened paste.

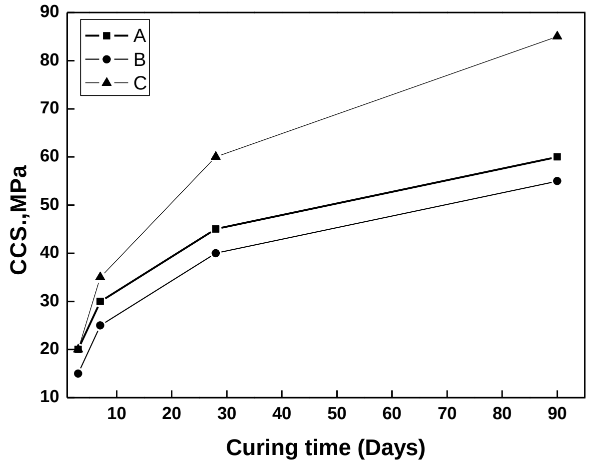

Fig.6, The mechanical strength of the hardened cement

Analysis of variance between groups to explore the effect of time on mechanical strength was applied. There was statistically significant difference between different times as determined by one-way ANOVA [F (3, 8) = 13), p = 0.002] as shown in Tables (16&17). The multiple comparison using Tukey (Table 18), revealed that the time 3 days is statistically significantly lower than 28 days, and 90 days. Also, it shown that the time 7 days is statistically significantly lower than 90 days. However, no significant difference between the 3 days and 7 days between 7 days and 28 days.

Table 16, ANOVA for mechanical strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3996.917	3	1332.306	13.257	.002
Within Groups	804.000	8	100.500		
Total	4800.917	11			

Table 17, Descriptive statistics for mechanical strength

					95% Confidence Interval for Mean			
	N	Mean	Std. Deviation	Std. Error	Lower Bound	Upper Bound	Minimum	Maximum
3 days	3	18.6667	3.21455	1.85592	10.6813	26.6521	15.00	21.00
7 days	3	30.0000	5.00000	2.88675	17.5793	42.4207	25.00	35.00
28 days	3	48.3333	10.40833	6.00925	22.4776	74.1891	40.00	60.00
90 days	3	66.6667	16.07275	9.27961	26.7397	106.5936	55.00	85.00
Total	12	40.9167	20.89131	6.03080	27.6430	54.1904	15.00	85.00

IV. CONCLUSION

The results of these study show that the use of water treatment plant sludge as a partial replacement of portland cement is feasible and promising, since they can be used directly without additional grinding and at a relatively low firing temperature. It can be assumed that this sludge could be a valuable renewable resource of raw materials in cement manufacturing. The obtained results show that adding 30 % of calcined sludge generally increases the chemically combined water, bulk density and mechanical strength of the hardened cement, but decreases the apparent porosity of the samples leading to an improvement of the densification and hence the mechanical properties and 30 % addition of dry WTPS resulted in increased water absorption and decrease in compressive strength and bulk density respectively. Studying the effect of different proportions of added sludge and calcined sludge as a partial replacement of ordinary portland cement, the calcination of WTPS at 700 ^{o}C resulting in formation of minerals that improves the quality of cement such as an increase in the concentration of calcium aluminate and calcium oxide during the incineration of sludge, then it can be considered as a primary or secondary admixture and provide environmentally friendly disposal methods for the sludge wastes. Thus, the reuse of calcined WTPS as construction material provides a good opportunity to reduce depletion of resources. As we know the cement is one of the most widely use construction materials and is rather expensive as compared to other materials. The construction cost can be directly cut down by replacing the cement with calcined WTPS. Thus, the use of calcined WTPS as cement replacement can greatly reduce the waste problem and save energy.

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APPENDICES

Multiple Comparisons

Table 6. Tukey HSD for Combined water of hardened pastes

	(I) water of hardened cement		(J) water of hardened cement	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
							Lower Bound	Upper Bound
dimension2	A	dimension3	B	.98000	1.69455	.835	-3.7512	5.7112
			C	-8.71000*	1.69455	.002	-13.4412	-3.9788
	B	dimension3	A	-.98000	1.69455	.835	-5.7112	3.7512
			C	-9.69000*	1.69455	.001	-14.4212	-4.9588
	C	dimension3	A	8.71000*	1.69455	.002	3.9788	13.4412
			B	9.69000*	1.69455	.001	4.9588	14.4212

*. The mean difference is significant at the 0.05 level.

Multiple Comparisons

Table 11. Tukey HSD for Bulk density of hardened pastes

	(I) Curing days		(J) Curing days	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
							Lower Bound	Upper Bound
dimension2	3 days	dimension3	7 days	-.0316667	.0224252	.526	-.103480	.040147
			28 days	-.0666667	.0224252	.069	-.138480	.005147
			90 days	-.1030000*	.0224252	.008	-.174813	-.031187
	7 days	dimension3	3 days	.0316667	.0224252	.526	-.040147	.103480
			28 days	-.0350000	.0224252	.449	-.106813	.036813
			90 days	-.0713333	.0224252	.052	-.143147	.000480
	28 days	dimension3	3 days	.0666667	.0224252	.069	-.005147	.138480
			7 days	.0350000	.0224252	.449	-.036813	.106813
			90 days	-.0363333	.0224252	.420	-.108147	.035480
	90 days	dimension3	3 days	.1030000*	.0224252	.008	.031187	.174813
			7 days	.0713333	.0224252	.052	-.000480	.143147
			28 days	.0363333	.0224252	.420	-.035480	.108147

*. The mean difference is significant at the 0.05 level.

Multiple Comparisons

Table 18. Tukey HSD for mechanical strength of hardened pastes

(I) Curing days	(J) Curing days	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound

dimension2	3 days	dimension3	7 days	-11.33333	8.18535	.541	-37.5457	14.8790
			28 days	-29.66667*	8.18535	.028	-55.8790	-3.4543
			90 days	-48.00000*	8.18535	.002	-74.2124	-21.7876
	7 days	dimension3	3 days	11.33333	8.18535	.541	-14.8790	37.5457
			28 days	-18.33333	8.18535	.192	-44.5457	7.8790
			90 days	-36.66667*	8.18535	.009	-62.8790	-10.4543
	28 days	dimension3	3 days	29.66667*	8.18535	.028	3.4543	55.8790
			7 days	18.33333	8.18535	.192	-7.8790	44.5457
			90 days	-18.33333	8.18535	.192	-44.5457	7.8790
	90 days	dimension3	3 days	48.00000*	8.18535	.002	21.7876	74.2124
			7 days	36.66667*	8.18535	.009	10.4543	62.8790
			28 days	18.33333	8.18535	.192	-7.8790	44.5457

*. The mean difference is significant at the 0.05 level.