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ORIGINAL CONTRIBUTION

Experimental Investigation and Classification of Wastewater Treatment Sludge as Pozzolan for Cement

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Abstract This paper presents an investigation conducted to evaluate wastewater treatment sludge for potential use as a cement extender or pozzolan. Two varieties comprising the digested wastewater sludge (DWWS) and activated wastewater sludge (AWWS) materials, were finely ground then calcined at 750 °C for 30 min. Subsequently, the calcined sludge waste materials were blended with ordinary Portland cement CEM I 52.5 N at 0, 10, 20, 30% DWWS or AWWS, and used to prepare mortar mixtures at proportions of 1:3:0.61 cement to sand to water. Various tests were conducted including workability, compressive strength, pozzolanic activity with lime, drying shrinkage and alkali-silica reaction. It was found that both varieties of the sludge waste materials, once blended with CEM I at 20% DWWS or 20% AWWS, satisfied the ASTM C618 criteria for Class C pozzolan, and may be classified as CEM II /A-P 32.5 N,R cement type of EN 197-1/SANS 50197-1. The overall performance of DWWS was relatively better than that of AWWS.

Keywords Pozzolans · Extenders · Wastewater sludge · Compressive strength · Alkali–silica reaction · Drying shrinkage

Introduction

Water treatment plants in South Africa discharge large volumes of sludge waste, ranging between 500 and 1300 tons per day. Bourgeois et al. [3] mentions that a water treatment plant may typically discharge approximately one hundred thousand (100,000) tonnes of sludge waste per year. The method of producing sludge through wastewater treatment (WTS) is typically done in two stages comprising:- (1) biological digestion which involves flocculation—clarification process carried out to separate the solid particles or sludge from liquid, and (2) further treatment of the liquid phase by employing sedimentation and filtration. WTS waste typically contains significant amounts of useful substances such as iron, calcium and silicon. However, actual composition of sludge waste depends mainly on the source and quality characteristics of wastewater or sewage. Most WTS waste is disposed—off to landfills, leading to environmental pollution typically associated with odours, leaching of toxic elements, soil and groundwater contamination. Such usage of landfills for waste disposal, also contributes to the shortage of land needed for agricultural farming and housing [27].

An alternative measure being explored worldwide to minimize the disposal of WTS to landfills, is incineration of the waste to produce sludge ash [24, 30]. However, incineration is an expensive undertaking owing to the high energy use along with the cost of associated infrastructure requirements, which in turn constrain practical employment of the technique. Developing countries that have a coastline, typically find it cheaper to dispose—off wastewater, sewage or sludge, directly into the ocean or sea. This undesirable method of waste disposal is apparently the most commonly employed worldwide [24]. However, environmental concerns of the modern era, demand the rethinking of waste disposal strategies and require the promotion of waste utilization as a

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resource. Accordingly, beneficial utilization of waste is being promoted through scientific research endeavours, towards mitigation of environmental pollution. Moreover, waste utilisation also reduces consumption of the limited natural resources used for industrial manufacturing of products such as cement etc.

Use of Sludge Waste Materials in Cementitious Systems

Over the past 20 to 30 years, various researches have been conducted to study WTS for use in cement. Several potential construction applications for WTS utilisation are listed in the literatures [13, 29], including use of the sludge waste as raw feed material for producing cement, as a pozzolan or extender for partial cement replacement, as fine aggregate filler, as an ingredient for making concrete bricks or blocks and for making lightweight foamed concrete products etc. [2, 33].

Gastaldini et al. [7] conducted an investigation involving incineration of WTS at various high temperatures of 400, 500, 600 °C for durations of 1 to 2 h or incineration at 700 °C for 30 min. The calcined sludge material obtained was blended with cement, then used in concretes of water to cementitious (w/cm) ratios = 0.35, 0.50 and 0.65. It was reported that calcination temperatures of 600 to 700 °C, produced sludge ash material that showed the best performance results. In a similar study, Naamane et al. [21] determined that at low calcination temperatures of 300 to 500 °C, not all absorbed water within sewage sludge (SWG) ash is removed, and that some particles remain unburnt. Consequently, SWG ash that was incinerated at low temperatures below 500 °C then blended with cement, gave mortar mixtures of lower strength. Incineration temperatures of 700 to 800 °C were found to produce SWG ash that gave optimal performance, with the 15% SWG blend with cement, exhibiting the best results. Juala et al. [8] reported that the incineration temperatures 800 to 900 °C, produced SWG ash that gave the best performance.

Plants used to treat drinking water produced sludge which was incinerated at 600 to 900 °C for 3 h, as examined by Tantawy [28]. The incineration temperature of 800 °C reportedly gave the most optimal enhancement of the material's pozzolanic characteristics.

Monzo et al. [18, 19] conducted investigations using mortars containing 15% or 30% SWG, prepared at aggregate / cement ratio = 3.0 and w/cm = 0.44. The mortar mixtures were cured at 40 °C, then measured to determine compressive and flexural strengths. The mortars containing SWG, showed higher results over those of the control. Although the SWG had a high sulphate content of 12.4% SO_3, it was concluded that compressive strength was not adversely affected.

It has been reported in some literatures that heavy metals, sulphates and more especially phosphorus, begin to adversely influence the performance responses of mixtures, once the SWG proportion in cement is between 6 to 11%, leading to increased setting time durations and strength reduction [13].

Phosphorus in SWG can be high, being typically about 12.6% P_2O_5. It is known from cement production literatures that P_2O_5 as low as 1.0%, can have severe effects on cement clinker reactions, owing to the tendency of phosphorus to form a solid solution with C_2S, in turn reducing the amount of C_3S formed [12, 22]. It is thus feared that high P_2O_5 content in cementitious systems containing SWG, could cause adverse effects comprising long setting times and low strength [32]. The SWG ash used in Chen and Poon's [4] study as pozzolan, had 9.72% P_2O_5 content. The compressive strengths and microstructural characteristics for mixtures containing up to 10% SWG, were not adversely affected.

Past Studies on Wastewater Treatment Sludge from South Africa

Hardly any research investigations have hitherto been done on South African wastewater treatment sludge (WWS), as pozzolanic material. Mathye et al. [15] studied WWS utilisation as sand replacement in concrete. Moreover, the proportions not exceeding 5% WWS used as sand replacement, were low and not consistent with the general need for utilisation of sludge waste in large quantities. A similar study was done by Mojapelo et al. [17], in which sludge waste was also utilized as sand replacement in concrete mixtures, but in this case, higher sand replacement proportions of up to 12.5% WWS, were employed. As mentioned already, no research literatures could be found on use of South African WWS as pozzolan or cement extender.

Objectives

In developing countries, the ongoing growth of urbanization guarantees the anticipated future increase in the generation of WWS. Already in urban cities, there is an enormous generation of WWS waste which is typically disposed—off to landfills. In this paper, calcined WWS was blended with CEM I 52.5N, following which mortars were prepared and used to conduct performance evaluation. Various tests were conducted including determination of particle size distribution, mortar flow, lime–pozzolan reactivity, compressive strength and drying shrinkage, along with alkali–silica reaction (ASR). Characterization studies were also done using scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS).

Experimental Study

Materials

Investigation involved two varieties of water treatment sludge materials comprising the digested wastewater sludge (DWWS) and the activated wastewater sludge (AWWS), both obtained from water treatment plants in Johannesburg, South Africa. Both varieties of the raw sludge materials had been sun—dried at the treatment plants, prior to collection for use in the present investigation.

DWWS was derived from the primary stage of WTS, wherein debris and grit i.e. sand and gravel etc. were screened out, then the remaining particles suspended in water, were allowed to settle in a sedimentation tank to form primary sludge with or without anaerobic digestion. AWWS emanated from the secondary treatment process wherein wastewater effluent from primary treatment was exposed to oxygen thereby allowing aerobic digestion by micro—organisms and bacteria that breakdown dissolved organic matter, in turn producing secondary or activated sludge and cleaner water effluent [16]. The secondary or activated sludge typically contains a higher concentration of micro—organisms, relative to that of primary sludge. The sludge materials were sampled randomly from their sun—drying beds, for use in the experiment.

To prepare DWWS and AWWS for blending with Portland cement, the raw sludge materials were first dried at 50 °C over 24 h period, then finely ground for 12 to 15 h using a laboratory ball mill. An electric furnace heating at 10 °C /min, was used for calcining the powder sludge materials at 750 °C for 30 min. Sun—dried, milled or calcined sludge waste materials are shown in Fig. 1 at the different preparation stages. The calcined sludge ash materials were blended with CEM I 52.5 N from PPC (Pty) Ltd, then mortars were cast for testing. The various cementitious materials used, are given in Table 1 showing the composition of each material. Tests comprising Blaine fineness and retention (%) on the 45 μm sieve, were done to measure fineness levels of the milled sludge waste materials (ASTM C430, 2017; ASTM C204, 2018).

The hydrometer method ASTM D422 (2007) was employed to determine particle size distribution curves of Portland cement, DWWS and AWWS. The analyses were done using the hydrometer type 152H. During the test, a 50 g sample was poured into the one—litre measuring cylinder, then tap water was added into the cylinder up to 980 ml mark, followed by addition of 10 mls of Chryso Premia 310 superplasticizer. The aqueous solution mixture was vigorously shaken, then additional water was poured into the cylinder, topping up the solution to 1000 mls. Readings were recorded at different time intervals for a period lasting up to 3 h. Upon completion of hydrometer measurements, the sediment material in the cylinder was washed onto the 45 μm sieve and used to determine the percentage retained.

Mixtures and Procedures

Mechanical properties were investigated using mortar mixtures prepared at 1 to 3 cement to sand ratio and 0.61 w/cm. The calcined sludge powder materials were incorporated in CEM I 52.5 N cement at proportions of 0, 10, 20, 30% DWWS or AWWS. Sallies Silica (Pty) Ltd provided the graded standard sand (ASTM C778, 2007) that was made by combining the different sizes 0.8 to 1.8 mm, 0.4 to 0.85 mm and 0 to 0.6 mm, at ratios of 4:1:3 respectively [5]. Mortar was first mixed slowly for 1 min, following which water was added slowly over 30 secs while mixing continued for 1 min, then high speed mixing was carried out for a further 1 min (ASTM C305, 2016). The flow test was done (ASTM C1437, 2020) immediately upon completion of mortar mixing.

The 50 mm mortar cubes were cast and cured in the water bath maintained at 40 °C. Compressive strength was tested at 3, 7, 28, 42 and 90 d (ASTM C109, 2019). The strength activity index (SAI) values were calculated at 7 and 28 d.

PAL test was conducted using mixtures prepared at 1:1:6 hydrated lime to sludge material to sand, and at 0.61 water to binder ratio. The lime—sludge mortar mixtures were cast then cured at 55 °C for 7 d, as per ASTM C311 (1996; 2013). Table 2 gives the various tests done including drying shrinkage and ASR etc. Also shown in Table 2 are the mixture quantities used.

(a) Sun - dried raw sludge (b) Milled raw sludge (c) Calcined sludge ash

Fig. 1 Illustration of preparation stages showing (**a**) sun—dried raw sludge, (**b**) milled raw sludge and (**c**) calcined sludge ash

Table 1 Chemical compositions of the cementitious materials used, comprising Portland cement (CEM I), digested wastewater sludge (DWWS) and activated wastewater sludge (AWWS)

Oxides and physical properties	CEM I 52.5N	DWWS		AWWS	
		Raw (%)	Calcined (%)	Raw (%)	Calcined (%)
Al_2O_3 (%)	3.45	5.26	14.01	3.77	12.75
BaO (%)		0.07	0.18	0.06	0.19
CaO (%)	68.5	2.86	7.92	3.04	9.96
Cr_2O_3 (%)	0.0285	0.36	1.00	0.07	0.49
Fe_2O_3 (%)	3.86	3.99	10.31	3.76	12.28
K_2O (%)	0.272	0.56	1.52	0.46	1.56
MgO (%)	1.37	1.18	3.23	1.09	3.75
MnO (%)		0.68	1.77	0.35	0.94
Na_2O (%)	0.242	0.17	0.48	0.18	0.67
NiO (%)		-	0.06	-	0.09
P_2O_5 (%)		5.32	14.07	4.30	14.34
SiO_2 (%)	17.8	15.47	41.34	11.47	38.68
SO_3 (%)	3.41	-	0.29	0.06	0.38
TiO_2 (%)	0.528	0.38	1.00	0.32	1.07
$Na_2Oe = Na_2O + 0.658\ K_2O$ (%)	0.42		1.48		1.70
LOI (%)	-	63.34	1.59	70.81	1.50
Density (g/cm^3)	3.15		2.84		2.87
Blaine fineness (m^2/kg)			504		1069
Retention on 45 µm sieve (%)			1.13		0.75

Table 2 Mortar mixtures of the tests conducted

	Mix ID	Mix ingredients					Tests
		Sample size (mm)	CEM I 52.5 N (g)	DWWS or AWWS (g)	Silica sand (g)	Water (g)	
DWWS or AWWS	0	50 mm cubes	586	–	1758	360	Workability, compressive strength
	10	50 mm cubes	527	59	1758	360	Workability, compressive strength
	20	50 mm cubes	469	117	1758	360	Workability, compressive strength
	30	50 mm cubes	411	176	1758	360	Workability, compressive strength
DWWS or AWWS	0	25 × 25 × 285	586	0	1758	360	Drying shrinkage
	20	25 × 25 × 285	469	117	1758	360	Drying shrinkage
DWWS or AWWS	0	25 × 25 × 285	400	0	900	240	Alkal–silica reaction
	20	25 × 25 × 285	312	78	879	240	Alkalil–silica reaction

For drying shrinkage monitoring, the samples employed were the 25 × 25 × 285 mm prisms made at 1 to 3 cement to sand ratio and 0.61 water to cementitious ratio. After moist—curing for 7d, the samples were exposed to air. Changes in length measurements have been recorded weekly at 0, 7, 14, 28, 35, 42 and 49 d.

Small mortar prisms 25 × 25 × 285 mm for testing of ASR, were cast using the reactive aggregate, greywacke from Peninsula, Western Cape in accordance with ASTM C1260 (2014) and SANS 6245 (2006) (Table 2). After storing the prisms in 1 N sodium hydroxide solution at 80 °C, expansion measurements were done at 7, 14, 21 and 28 days.

Results and Discussion

Characterization of the Sludge Materials

Fineness Along with Particle Size Distribution

Interestingly, for same duration of grinding i.e. 12 to 15 h, there was a remarkable difference in fineness levels of DWWS and AWWS, with the latter giving Blaine fineness of 1069 m^2/kg which is about twice 504 m^2/kg of the former (Table 1). These observations are consistent with the results determined based upon retention on 45 µm sieve, i.e. AWWS gave 0.75% retention on the sieve, which is about one—half of 1.13% retention observed for DWWS (Table 1). Evidently, AWWS appeared to be softer and was easier to grind than its counterpart DWWS. Further research is needed to determine the mineralogical factors responsible for the observed large difference in grindability behaviours of the two sludge material varieties.

The significant difference in grindability tendencies of the two sludge varieties, is also evident in their PSD curves shown in Fig. 2. Comparing the PSD curves of DWWS and AWWS, it can be seen that the latter gave a higher proportion of fine particles falling within 0 to 1.5 mm size range, but a relatively lower amount of coarse particles of 1.5 to 5.0 mm sizes. In contrast, the PSD curve of DWWS was closely similar to that of Portland cement CEM I. Fine particles typically promote reactivity and may act as filler material. However, very fine particles may also coagulate during mixing, which may necessitate using dispersants such as superplasticizers.

The sludge waste varieties DWWS and AWWS had relative densities of 2.84 and 2.87 respectively, which values are similar to those of some common pozzolans.

Influence of Calcination on Composition

The raw DWWS and raw AWWS materials generally had similar chemical compositions, with both varieties giving high values of loss on ignition (LOI) comprising 63.34% and 70.81% respectively. Specifically, however, the raw AWWS had relatively lower SiO_2 /Al_2O_3 content levels of 11.47% /3.77% compared to 15.47% /5.26% of raw DWWS. Either way, these content levels of SiO_2 and Al_2O_3 in the raw sludge materials, are too low and of insignificant influence on strength development in cementitious systems, as observed later in Sect. "Compressive strength results". As such, it can be deduced that the raw sludge waste materials may not be of much use as pozzolans.

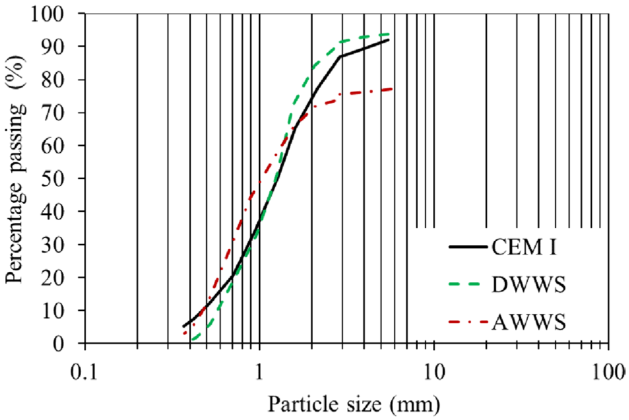


Fig. 2 Particle size distributions of ordinary Portland cement (CEM I 52.5 N), digested wastewater sludge (DWWS) and activated wastewater sludge (AWWS)

Upon calcination of the raw sludge materials, however, their chemical constituents were strongly altered and their characteristics were enhanced towards pozzolanic reactivity. Table 1 shows that the content levels of alumina, silica and iron in the sludge materials, all increased significantly from 4–5% Al_2O_3, 11–15% SiO_2 and 4.0% Fe_2O_3 before calcination, to 13–14% Al_2O_3, 38–40% SiO_3 and 10–12% Fe_2O_3 after calcination. These chemical compositions of the calcined DWWS and calcined AWWS materials, are similar to those of natural pozzolans such as volcanic ash typically composed of 14–15% Al_2O_3, 42–47% SiO_3 and 13–14% Fe_2O_3 [6, 11], zeolite or metakaolin [1, 26].

The most significant effect of calcination on composition, was enhancement of $SiO_3 + Al_2O_3 + Fe_2O_3$ content from 24.7% /19% in raw DWWS /AWWS before calcination, to the respective 65.7% /63.7% after calcination. The calcined DWWS /AWWS materials both meet the minimum requirement of $SiO_3 + Al_2O_3 + Fe_2O_3 \geq 50\%$ for Class C pozzolan (ASTM C618, 2015). Also, the LOI values comprising 1.59% /1.50% of calcined DWWS /AWWS, are very low and well below the specified maximum limit of 6%. The foregoing observed thermally—induced compositional changes also show that calcination was highly effective in decomposing organic matter and in removing absorbed water, both of which formed large bulk quantities in the raw sludge materials, as indicated by the initially high LOI values 63.34% /70.81% of the raw DWWS /AWWS.

It is known that sulphates and phosphates can be major constituents in sludge waste materials (Sect. "Use of sludge waste materials in cementitious systems"). In the present study, the values 0.29% /0.38% SO_3 of calcined DWWS / AWWS (Table 1), are less than the specified limit of 5% SO_3 (ASTM C618, 2015). It may thus be deduced that there would be a low or no risk of internal sulphate attack in cementitious systems containing the sludge waste materials.

Scanning Electron Microscopy

Specimens of calcined DWWS /AWWS were prepared for SEM examination, done using the TESCAN VEGA3SEM coupled with EDS. Figure 3 gives microstructural features of the DWWS and AWWS powder materials. Both images of Fig. 3a, b show the particles exhibiting some vesicular features, which may partly explain the lower relative density values 2.84/2.87 of DWWS /AWWS. It is evident that particles of the sludge waste material were angular, which is consistent with similar reports in the literatures [17, 34]. The observed angular shape of sludge particles, arises from the attrition during grinding which randomly fractures the material in an irregular manner. Coagulation of particles can be seen in both micrographs of Fig. 3a, b, an observation that

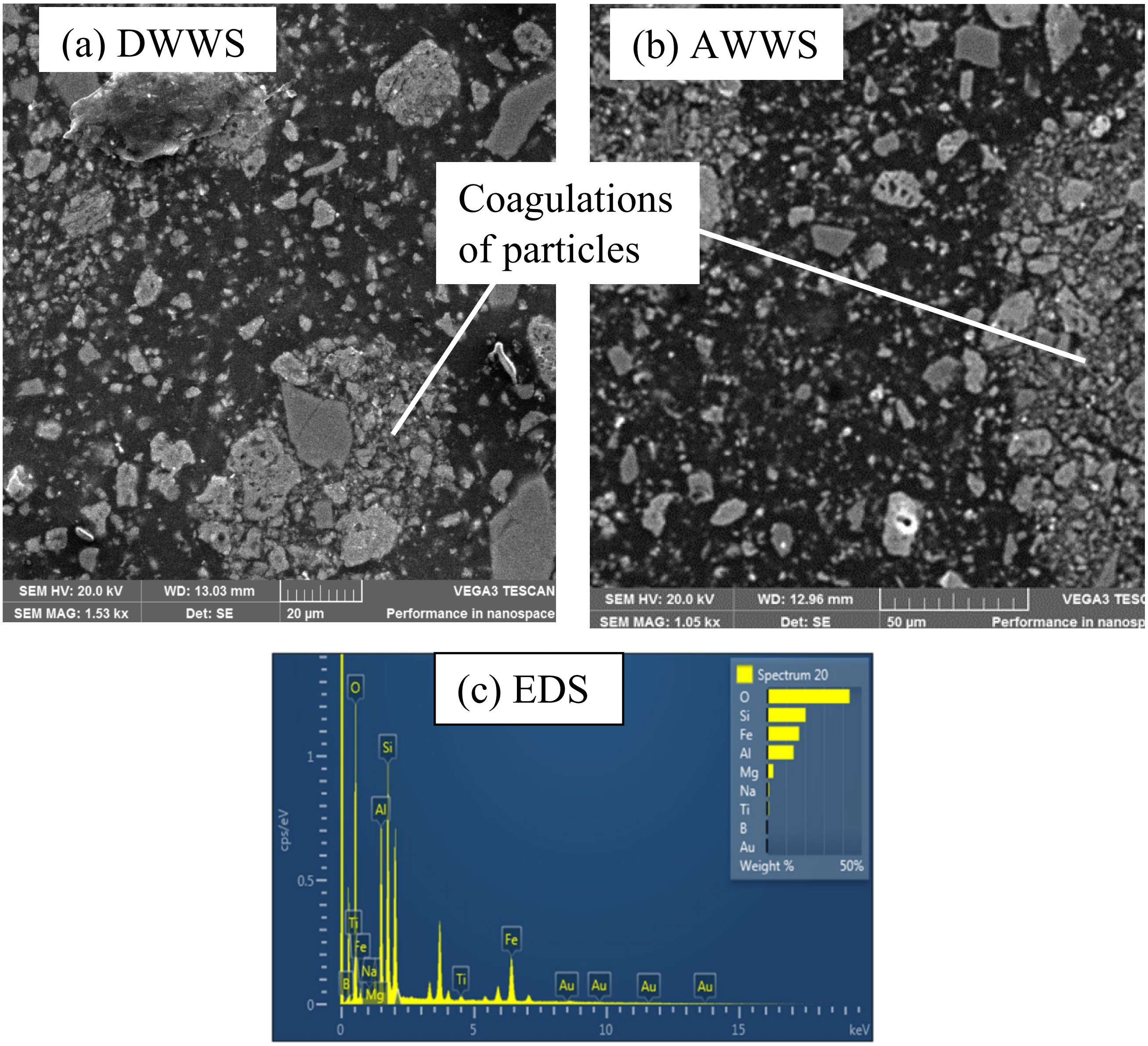


Fig. 3 SEM analysis of (**a**) digested wastewater sludge (DWWS), (**b**) activated wastewater sludge (AWWS) and (**c**) energy dispersive spectroscopy (EDS) analysis

may be attributed to high fineness of the powder materials. EDS of Fig. 3c shows that the main chemical constituents of the calcined sludge materials were aluminosilicates, an observation which is characteristic of pozzolans, and is also consistent with the chemical analyses results given in Table 1.

Pozzolanic Activity with Lime

The PAL mortar mixtures of proportions 1:1:6 lime to DWWS /AWWS to sand, prepared at 0.61 water /binder ratio then cured at 55 °C ("Mixtures and Procedures" section), gave 7—d strength values of 7.23 /7.87 MPa, respectively. Clearly, both results exceed the minimum value of 5.5 N/mm^2 (ASTM C618, 1989). Generally, for common pozzolans to be effective, their fineness values should be similar to or greater than cement fineness which is typically 300 to 500 m^2/kg. In this study, the fineness levels for DWWS /AWWS were 504 m^2/kg and 1069 m^2/kg respectively, which are both quite high. Similarly, the percentage values of DWWS and AWWS retained on 45 μm sieve, were respectively 1.13% and 0.75%, which are far below the maximum limit of 34% specified in ASTM C618 (2015).

Workability

Figure 4 shows that flow results increased correspondingly with rise in the amount of DWWS or AWWS incorporated into cement. Interestingly, the flow results of DWWS mortars were higher than those of the corresponding AWWS mixtures. Moreover, increases in workability values of DWWS mortars over those of AWWS mixtures, became greater as sludge ash content increased. Flow results of mortar mixtures containing the sludge ash materials, increased from 143.3 mm of the control to 156.7 /150.1 mm for mortars containing 30% DWWS /30% AWWS, which represent 9.4% /4.7% corresponding increases in workability levels. Mahutjane et al. [14] reported similar findings, wherein sludge that was calcined at varied temperatures of 550 to 850 °C was used to prepare geopolymer mortars. It was reported that sludge calcined at higher temperatures gave correspondingly higher workability values of mortars, an effect they attributed to high fineness of the waste material with rise in incineration temperature. It is also reported in Monzó et al. [20] and Pan et al. [23] that higher fineness of sludge ash leads to increase in workability. In contrast, other literatures show decrease in workability upon use of sludge in cement and concrete [9, 10, 25, 31]. It is evident that high fineness levels of DWWS and AWWS seemingly had an overriding effect over other factors, accordingly leading to the observed increase in workability. However, further research is needed to fully evaluate this aspect.

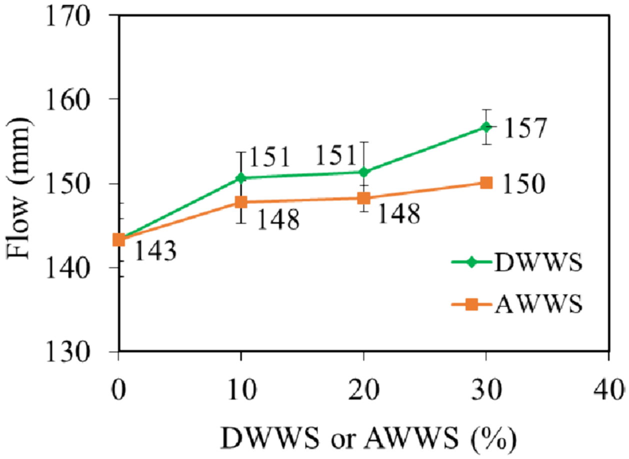


Fig. 4 Workability results of mortars containing digested wastewater sludge (DWWS) and activated wastewater sludge (AWWS)

It may be recalled that AWWS had a higher amount of fine particles relative to DWWS or CEM I ("Characterization of the sludge materials" section). Fine particles not only have higher water demand, but are typically more angular and also serve as filler material, thereby enhancing the interlocking mechanism between different particles. A combination of the foregoing factors may explain the lower workability values of the mixtures containing AWWS relative to those of DWWS mortars.

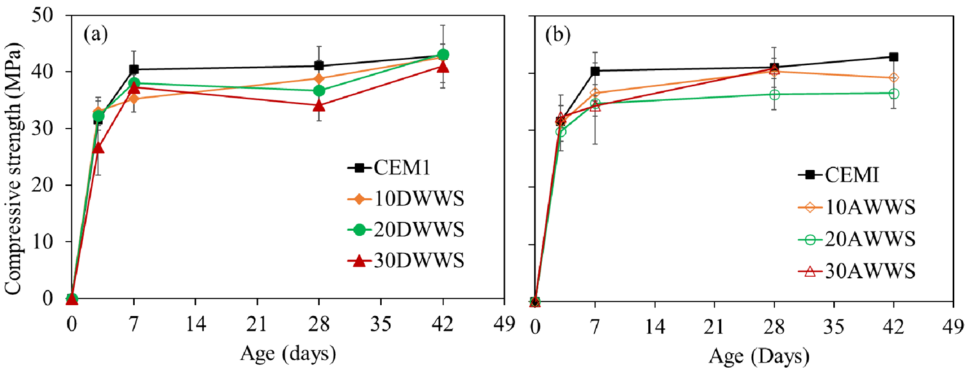
Compressive Strength Results

Strength results show that calcination of the sludge materials employed in the present study, was a necessary thermal treatment process that activated pozzolanic characteristics of the waste by-products. For example, an attempt was made to prepare mortar mixtures using raw DWWS or raw AWWS. It was found that mortar mixtures containing the raw sludge ash materials, were unable to achieve strength development even when cured at the elevated temperature of 40 °C. Based on the foregoing observations, no further experimental investigations were done using the raw sludge ash materials. Rather, the investigations presented in this paper were thus done using calcined DWWS or calcined AWWS.

Strength Development

Figure 5 gives the strength development curves of mortar mixtures containing 0 to 30% calcined DWWS or AWWS. At 3 d, the 20% DWWS or 20% AWWS attained higher or similar strength values as the control, giving 31 to 33 MPa. Beyond 7 d of curing, however, separation of the curves occurred with mortars containing the sludge ash materials, expectedly giving lower strengths, relative to

Fig. 5 Compressive strengths for mortars containing (**a**) digested wastewater sludge (DWWS) and (**b**) activated wastewater sludge (AWWS): CEM I—ordinary Portland cement CEM I 52.5 N mortar



corresponding values of the control. However, the strength growth trends for DWWS and AWWS mortar mixtures, were quite different from that of the control CEM I. Evidently, strength gain for the control CEM I mixture mostly occurred within the first 7 d, with only minor further growth occurring thereafter. Indeed for the control mixture, about 90 to 98% of the 28—d strength was attained within the first 7 d. In contrast, mortars containing DWWS or AWWS, showed relatively lower strength values within the first 7 d, but attained progressively higher gains during late strength development, which is typical of pozzolanic behaviour, as depicted by results of both mortars containing DWWS or AWWS.

Clearly, replacement of cement with 10% DWWS or 10% AWWS exhibited strong long—term strength performance levels that were generally close to that of the control. It is estimated that partial cement replacement with 10% sludge ash, would contribute a cost saving of 5% to 10%, which can be a significant benefit towards affordability in applications such as housing construction, for example.

Strength Activity Index

Table 3 gives the SAI values determined. SAI is the proportion of compressive strength attained by mixtures containing pozzolanic materials, relative to the corresponding value of control. Table 3 shows that the 7—d and 28—d SAI values were all between 83.0 to 99.6%, and thus meet the criterion of 75% minimum value (ASTM C618, 2015). As expected, SAI generally decreased with increase in the proportion of DWWS /AWWS incorporated, an observation partly attributed to the dilution effect due to reduction in clinker content upon partial replacement of cement with the sludge waste materials. It may also be noted that the SAI values of DWWS and AWWS, were generally similar.

Table 3 Values of strength activity index

Cement or sludge ash	Proportion of sludge ash (%)	Compressive strength (MPa)		Strength activity index, SAI (%)	
		7 d	28 d	7 d	28 d
CEM I	0	40.41	41.01	–	–
DWWS	10	35.26	38.8	87.3	94.6
	20	38.02	36.67	94.1	89.4
	30	37.27	34.08	92.2	83.1
AWWS	10	36.56	40.31	90.5	98.3
	20	34.68	36.29	85.8	88.5
	30	34.3	40.84	84.9	99.6

Durability Performance

Drying Shrinkage Results

Results of drying shrinkage of mortars containing 20% DWWS or 20% AWWS, are given in Fig. 6. It is evident that mixtures containing the sludge ash materials generally showed relatively enhanced levels of drying shrinkage, compared to that of the control mortar. The DWWS and AWWS mortars gave 28—d shrinkage results of 0.084% and 0.113% respectively, which are slightly higher than the corresponding 0.077% value of the control CEM I mortar. Evidently, the 20% DWWS and CEM I mortar mixtures, both met the specified maximum 28—d drying shrinkage limit of 0.08%, while 20% AWWS mortar failed to meet the criterion as it gave higher shrinkage of 0.113% (ASTM C596, 2018). Evidently, proportions higher than 20% sludge content would give higher shrinkage value(s) exceeding the prescribed maximum limit.

Alkali–Silica Reaction

The alkali levels comprising 1.48%/1.70% Na_2Oe of DWWS/AWWS were much higher than the 0.42% Na_2Oe content of CEM 1 (Table 1). However, the high alkali levels of the sludge ash materials may not be of major concern, considering that the pozzolan Na_2O and K_2O usually don't participate in ASR. But in some cases such as the volcanic tuff reported in Ekolu et al. [6], alkalis in pozzolans may promote ASR, hence the necessity to test the candidate materials experimentally rather than make determinations based on chemical compositions only. Indeed, Fig. 7 shows that both sludge ash varieties were effective in reducing ASR attack, with DWWS giving better performance than AWWS.

The control CEM I mixture gave a 14—d expansion of 0.163% which is above 0.1% maximum recognized for

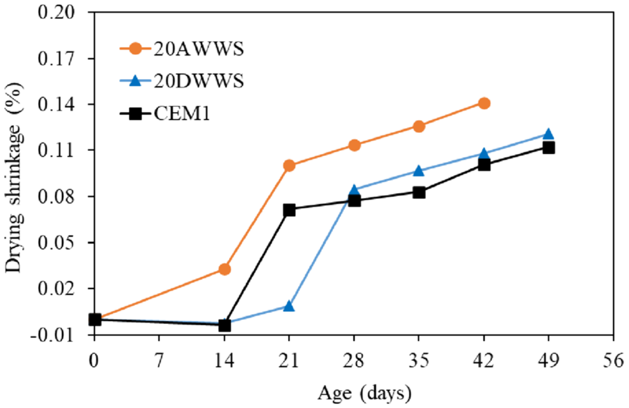


Fig. 6 Results of drying shrinkage mortars containing digested wastewater sludge (DWWS) or activated wastewater sludge (AWWS)

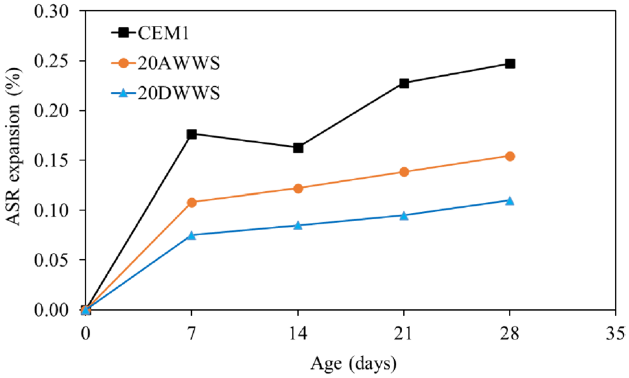


Fig. 7 Alkalil–silica reaction (ASR) expansions of mortars containing digested wastewater sludge (DWWS) or activated wastewater sludge (AWWS)

non—expansive systems. These results confirm the high reactivity of greywacke aggregate that was used to prepare ASR mortar mixtures. Mortars containing 20% DWWS and 20% AWWS gave lower 14—d expansions of 0.085% and 0.122%. Again, the effectiveness of DWWS in controlling ASR was greater than that of AWWS, as similarly observed for the drying shrinkage results ("Drying Shrinkage Results" section).

Classification of the Wastewater Treatment Sludge Waste Materials

The classification requirements for artificial pozzolans comprising natural pozzolans and fly ash, are specified in ASTM C618 standard which provides various criteria for evaluating quality and performance characteristics of the extenders. Class N category is exclusively reserved for natural pozzolans, while Class C and Class F categories each gives the criteria for classification of different fly ash types.

The selection of 20% sludge content as the suitable proportion for cement replacement, was based on comprehensive consideration of the ASTM C618 requirements given in Table 4. The maximum proportion of sludge content that gave results meeting the specified requirements, was selected to be the suitable dosage. The results discussed in section "Drying Shrinkage Results" section show that proportions higher than 20% sludge content would fail drying shrinkage requirements.

Table 4 compares the results of mixtures containing 20% DWWS or 20% AWWS against the criteria for each class category. It is evident that both varieties of sludge waste meet the requirements for their classification as Class C pozzolans.

The foregone results show that sludge waste materials that were investigated, exhibited adequate performance when incorporated into cement at the proportion of 20% DWWS or 20% AWWS. The 20% DWWS mortar gave 3/7—d compressive strength values of 32.3/38.0 MPa, while the corresponding results for 20% AWWS mortar were 31.2/34.7 MPa respectively. Evidently, the CEM I/20% DWWS or CEM I/20% AWWS blends generally satisfied the various criteria for classification of the composites as Class C pozzolan and CEM II/A-P 32.5 N, R cement (ASTM C618 and EN 197-1).

Conclusions

The foregone study evaluated wastewater treatment sludge materials for potential use as pozzolans. Two varieties comprising, the digested wastewater sludge (DWWS) and activated wastewater sludge (AWWS) materials were milled,

Table 4 Evaluation of wastewater treatment sludge materials for classification (ASTM C618, 2015)

	ASTM C618			Calcined DWWS (20%)	Calcined AWWS (20%)
	Class N	Class F	Class C		
$SiO_2 + Al_2O_3 + Fe_2O_3$, minimum (%)	70.0	70.0	50	65.7	63.7
SO_3, maximum (%)	4.0	5.0	5.0	0.29	0.38
Loss on ignition, maximum (%)	10.0	6.0	6.0	1.59	1.50
Fineness, by retention on 45 µm sieve, maximum (%)	34.0	34.0	34.0	1.13	0.75
Strength activity index: at 7 d, minimum (%)	75	75	75	94.1	85.8
: at 28 d, minimum (%)	75	75	75	89.4	88.5
Lime-pozzolanic activity, minimum (MPa)	5.5*	5.5*	5.5*	7.23	7.87
Water demand, max (% of control) or flow increase for 20% sludge ash	115	105	106	106	103
ASR expansion for test mix relative to that of control at 14 d, maximum (%)	100	100	100	68.2	98.2
Mortar drying shrinkage at 28 d, difference over control (%)	0.03	0.03	0.03	0.007	0.036

*Based on ASTM C618 (1989)

calcined then blended with CEM I 52.5 N cement. Mortar mixtures were prepared for performance evaluation.

It was found that both calcined sludge waste materials comprising DWWS and AWWS can be classified as Class C pozzolans, as per ASTM C618. Blends of 20% sludge waste materials with cement, also fulfilled the requirements for CEM II/A-P 32.5 N,R cement composite of EN 197–1.

As the amount of sludge waste material in the mixture increased, workability also increased. The blend of 20% DWWS or 20% AWWS with Portland cement, exhibited adequate strength development, adequate drying shrinkage behaviour and effective mitigation of alkali–silica reaction. DWWS consistently exhibited a relatively better overall performance than AWWS.

P_2O_5 content was significantly high in both sludge waste varieties. Further investigation is needed to assess possible adverse effects of the phosphorus agent, on cement and concrete properties.

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Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

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