

Enhancing the Pozzolanic Reactivity of Spent Bleaching Earth Ash (SBEA) in Binary Blended Cement Mortar through Calcination

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The effects of calcination on spent bleaching earth ash (SBEA) were investigated in terms of its contribution to pozzolanic reactivity as well as the strength of blended cement mortar when used as partial cement substitution. SBEA waste, which is plentiful in the global stock, showed some potential as pozzolanic material to reduce reliance on the traditional clinker cement. However, actual research on its use as cement replacement and how it may benefit from calcination was still very limited. This paper can conclude that SBEA contains sufficient silica and alumina oxides as well as satisfactory levels of pozzolanic reactivity to be classified Class N natural pozzolan. By calcining SBEA up to a temperature of 700 °C, its pozzolanic reactivity was improved due to the amorphisation of its contents, leading to an increase in the allowable level of replacements from 30 to 40 % as well as the peak strength achieved. These findings allowed calcined SBEA to be used in greater volume in cement mortar compared to an untreated SBEA. It is also worth noting that an excessively high calcination temperature around 900 °C was found to be detrimental to both the pozzolanic reactivity and strength development of cement mortars.

Keywords: spent bleaching earth ash; natural pozzolan; calcination; cement mortar

I. INTRODUCTION

Spent bleaching earth ash (SBEA) is a by-product from the edible oils refining industry. In its original form, it is known as Fuller's earth or bleaching earth which are essentially highly absorbent clay such as bentonite. Bleaching earth is used to remove pigmentation and impurities from raw processed oils, resulting in waste material called spent bleaching earth (SBE) which contains high amounts of residual oil (Loh *et al.*, 2013). This residual oil would then be extracted further using the Soxhlet process as bio-diesel (Huang & Chang, 2010) and the resultant SBE is used as fuel in the boiler before ending up as SBEA (Figure 1).

Bentonite is a montmorillonite-type clay (Sutherland, 2014) with a 2:1 layered structure comprising one octahedral sheet of alumina sandwiched in between two sheets of silicate (Fernandez *et al.*, 2011). This means montmorillonite would naturally contain high amounts of

silicate oxides that would give it good pozzolanic reactivity for use as cement replacement. However, the sources of montmorillonite are seldom pure and may contain traces of albite, cristobalite and quartz (Fernandez *et al.*, 2011). Past research has also found that use of pozzolanic material has 'filler' effect which improves particle packing within the microstructure of the concrete (Fernandez *et al.*, 2011).

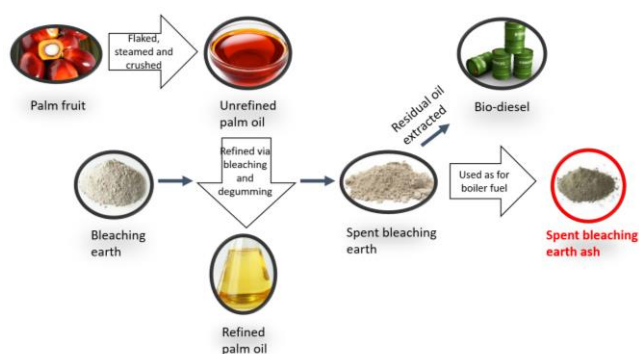
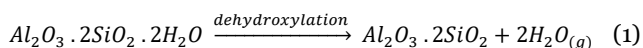


Figure 1. Production of SBEA

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Pozzolanic replacement in binary blended cement mortar (where part of the cement content is replaced with pozzolan) are very common these days such as the use of ground pumice powder (Karataş *et al.*, 2017), sugar cane bagasse ashes (Dembovska *et al.*, 2017) and metakaolin (Antoni *et al.*, 2012). However, the allowable magnitude of replacements is typically limited to under 30 % of the weight of cement so as to maintain practical working strength. To this end, activation of the pozzolan has been shown to improve its pozzolanic reactivity, and indirectly, its strength as well (Cordeiro *et al.*, 2019). Typically forms of activation include mechanical, thermal and chemical. Mechanical activation is very common and typically involves grinding the pozzolan into finer particles and hence larger surface area for reaction. Thermal activation, on the other hand, involves heating the pozzolan up to very high temperatures, in the range of 300 to 900 °C, called calcination which removes volatile substances and oxides a portion of its mass.

A very popular example where calcination was used to improve the pozzolanic reactivity of pozzolan is where highly amorphous metakaolin is produced by calcining kaolin clay (China clay). This process dehydrates and dehydroxylates the kaolin (Bellotto *et al.*, 1995), represented by the following reaction:



The level of de-hydroxylation depends on the heating condition such as heating temperature which will directly influence the improvement to pozzolanic reactivity. It has been found that reactivity of pozzolan increases with calcination at 700 °C (Vizcayno *et al.*, 2010) although complete de-hydroxylation of kaolin can occur anywhere between 650 to 850 °C (Wan *et al.*, 2017), depending on particle size. The higher end temperatures are only achievable with finer pozzolans level as these allowed greater de-hydroxylation (Ferreiro *et al.*, 2019). On the other hand, excessive heating past this range causes sintering and re-crystallisation of kaolin in the form of mullite which is detrimental to pozzolanic reactivity and strength (Ondro *et al.*, 2019). The duration of calcination has also been studied on paper mill sludge as pozzolan and it was found 2 hours produced the most reactive pozzolan (Jang *et al.*, 2018).

Besides increasing pozzolanic reactivity, calcination also improved workability through the slight reduction in the specific surface area of the transformed clay particles (Ferreiro *et al.*, 2019). The laboratory research into calcination was also found to be in good agreement with trials carried out in the industry (Almenares *et al.*, 2017).

This paper will investigate how calcination can be applied to SBEA and it affects the mechanical performance of cement mortar when used as partial cement replacement.

A. Materials and Preparation

The main focus of the experimental regime involved testing of cement mortar prepared using ordinary Portland cement (OPC), fine aggregates, water and SBEA as partial replacement of the OPC. For the OPC, CEM II 32.5 N conforming to BS EN 197:2014 was purchased commercially in bags of 50 kg from Cement Industries Sabah in Sabah, Malaysia. SBEA, the main test material used in the experimental regime, was supplied by Eco Oils Sdn. Bhd. based at Lahad Datu, Sabah in Malaysia. The SBEA powder was packed in thick polyethylene bags of approximately 5 kg each. Washed river sand, graded between 1.18 mm and 150 microns, from Kota Belud in Sabah was used as fine aggregates in the cement mortar samples. The physical and chemical properties of these materials are presented in Results and Discussion section A. All materials were pre-dried in the oven at 100 °C for 24 hours prior to use.



Figure 1. Calcination of SBEA in muffle furnace

This study includes investigation on the effect of calcination on SBEA at three different temperatures, namely 500 °C (SC500), 700 °C (SC700) and 900 °C (SC900) and to assess its impact when used as cement replacement in mortar. To carry out the calcination, approximately 50 g of pre-dried SBEA powder were placed in a porcelain crucible

and then heated using a muffle furnace (Figure 2) for 2 hours.

B. Test Methodology

The chemical composition of all three component materials were analysed using X-ray fluorescence (XRF) techniques using the Malvern Panalytical Epsilon 1 which is a fully integrated energy dispersive XRF analyser that consists of a spectrometer, computer and analysis software. XRF allows the identification of the amount of silicate and alumina oxides (SiO_2 and Al_2O_3) that contributes to strength as well as the unwanted sulfur trioxide (SO_3) that can compromise the performance of cement through by causing expansion in its lime and sulfate content. A loss on ignition (LOI) test was also carried out using a muffle surface at 1000 °C to determine the amount of unburnt volatiles contained within. The chemical composition of the SBEA, OPC and fine aggregates are shown on Table 3.

The mineral phases and crystal structure were characterised using X-ray diffraction (XRD) on a Rigaku Smartlabs diffractometer with Cu K α radiation operated at wavelengths of $k_1 = 1.45059$, $k_2 = 1.54441$ and a scanning mode of 2 theta from 3 to 85 °. XRD captures the intensity of the reflected ray to quantify the level of crystallisation of SBEA as it undergoes calcination at various temperatures.

The test samples also underwent particle size study using laser diffraction method to determine its sizes and surface area. For the fine aggregates which are generally coarser than 75 microns, mechanical sieving method was used instead.

The pozzolanic reactivity of SBEA was assessed based two different methods. The first was the strength activity index method detailed in ASTM C618 where cement mortar cubes containing 20 % pozzolanic replacement had to meet at least 75 % of the control specimen compressive strength. The second method uses thermogravimetric analysis (TGA) to quantify the residual calcium hydroxide (CH) remaining in cement mortar samples after 28 days of curing. The lesser the CH content would indicate higher pozzolanic activity in the sample.

Next, the mechanical effect of calcined SBEA on cement mortar cubes was assessed based on its effect on the i) water requirement of fresh mortar, ii) compressive strength of

mortar cubes at ages 7, 28 and 56 days and iii) flexural strength of mortar prisms at 28 days.

The mix design for the mortar cubes was based off ('ASTM C109/C109M-02 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars', 2002) and summarised in Table 1. The mix design allows six cubes to be prepared but for this experiment, the mix was re-proportioned for nine cubes so that there are three cubes available for each testing age (7-, 28- and 56-day). The cube size for compressive strength testing was 50 x 50 x 50 mm whilst 40 x 40 x 160 mm prisms were used for flexural strength testing.

Both SBEA and sand were used in their oven-dried condition, and the water content in the mix were adjusted based on their respective rates of absorption and moisture content to obtain a saturated surface dry (SSD) equivalent.

Table 1. Mix design for six no. control mortar cubes

Material	Amount
OPC	500 g
Graded sand	1375 g
Water	242 mL

A total of 15 different test mixes were prepared; a control mix using only OPC, seven test mixes using untreated SBEA to replace OPC from 10 to 70% replacement and another seven test mixes using calcined SBEA to replace OPC from 10 to 70% replacement (Table 2).

The control mix was prepared based on a fixed amount of water, after adjusted for the absorption rate of the sand, as shown on Table 1. Workability of fresh cement mortar was assessed using the flow table test. The test mixes were then prepared using additional water required to achieve similar flow of that for control mix ($\pm 5\%$).

Table 2. Mix design for test mortar cube samples

Code	Specimen
MCTRL	Control mortar containing OPC
MSUxx	Test mortar containing untreated SBEA
MSC5xx	Test mortar containing calcined SBEA (500 °C)
MSC7xx	Test mortar containing calcined SBEA (700 °C)
MSC9xx	Test mortar containing calcined SBEA (900 °C)

After casting, the samples were allowed to harden for 24 hours under cover before being transferred into tank containing lime saturated water for curing. Both the compressive and flexural strength of mortars were determined using a universal testing machine (Figure 3) for an average strength of three replicates per test sample.

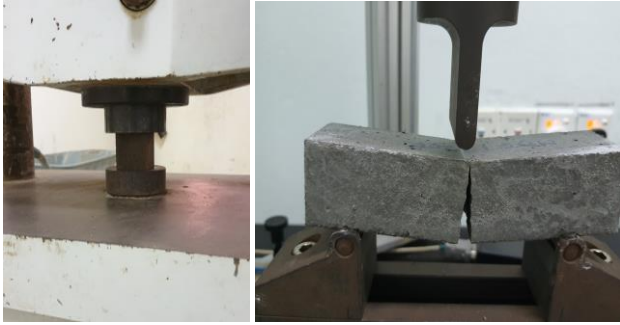


Figure 3. Compressive (left) and flexural (right) strength testing

II. RESULTS AND DISCUSSION

The test results presented in three separate sections; namely ‘A. Properties of Untreated and Calcined SBEA’ that focuses on the change in characteristics of SBEA itself as it undergoes calcination, ‘B. Pozzolanic Reactivity’ and ‘C. Strength Development of Cement Mortar Samples’ which will assess the impact of using calcined SBEA in cement mortar.

A. Properties of Untreated and Calcined SBEA

This section will delve into the physical and chemical properties of untreated and calcined SBEA and carry out comparison between the two.

1. Physical Properties

SBEA powder is supplied in very fine powder form, with grey-ish colour. It is smooth to the touch and disperses easily. On the surface, calcination was seen to change the pigmentation of the SBEA powder from its original greyish colour to an orange-ish hue (Figure 4). The higher the calcination temperature, the brighter orange it becomes.



Figure 4. Appearance of untreated vs calcined SBEA

SBEA particle sizes are typically coarser compared to OPC particles, with the mean particle size about twice as large (Table 3). Due to this, SBEA particles have just under half the specific surface area of OPC particles. Weight-wise, SBEA is just a little lighter compared than OPC.

Table 3. Physical characteristics of materials used

Material	Particle size (µm)			Specific surface area (m ² /kg)	Specific gravity
	d ₁₀	d ₅₀	d ₉₀		
Sand	272	345	1202	-	-
OPC	3.1	16.4	48.9	753.3	3.1
Untreated SBEA (SU)	8.1	36.2	100.1	347.1	2.8
Calcined SBEA 500 °C (SC500)	7.3	34.0	94.4	367.6	-
Calcined SBEA 700 °C (SC700)	7.0	33.1	93.4	379.4	-
Calcined SBEA 900 °C (SC900)	7.2	33.2	93.8	360.8	-

Calcination appears to have a very minor effect on the particle sizes of SBEA as presented in Figure 5. The most significant change came from a calcination temperature of 700 °C where mean particle sizes was reduced by 8.7 % whilst at the same time, increasing its specific surface area by 9.31 % but this is still relatively minor in the grand scheme of things. This was expected as similar findings were revealed in past research (Cordeiro *et al.*, 2009). However, extremely high calcination temperature (such as 900 °C) resulted in adverse effect where particle sizes increased slightly at the expense of surface area (Table 3).

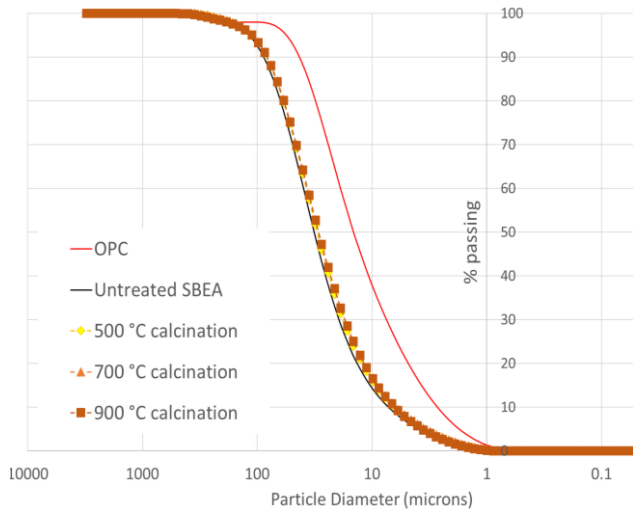


Figure 5. Particle size distribution of untreated and calcined SBEA

2. Chemical Properties

The chemical composition of the SBEA, OPC and fine aggregates are summarised in Tble 4. It can be seen that SBEA contains total oxides of 71.1 %, with a sulfur trioxide content of under 4 % and LOI of under 10 %, fulfilling the chemical requirements of Class N natural pozzolan as defined by (*ASTM C618–05 Standard Specification for Coal*

Table 4. Chemical composition of materials

Material		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	SO ₃	LOI
OPC		16.1	4.0	3.6	69.9	3.6	2.0
Sand		65.7	12.5	5.8	4.9	0	3.6
Untreated (SU)	SBEA	49.3	11.3	10.5	11.3	1.5	3.7
Calcined 700 °C	SBEA	-	-	-	-	-	2.5
Calcined 900 °C	SBEA	-	-	-	-	-	2.5

It is worth noting that the oxide content varies according to the SBEA stock or source as past research has shown that it could be both much higher (Rokiah *et al.*, 2019) or lower (Rahman *et al.*, 2020).

SBEA in its untreated form comprise a mixture of amorphous and crystalline components with a level of

Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete, 2005). Class N pozzolanic materials can exist as either raw or calcined pozzolans originating from diatomaceous earths such as cherts, shales, ashes.

However, despite meeting the LOI content requirement of Class N natural pozzolan, the volatiles content remained high at 3.7 %, which can be detrimental to workability and conductivity of cement mortar (Mu *et al.*, 2017). By calcining the SBEA, its LOI was reduced to 2.5 % which is beneficial in reducing the impact caused by the volatile contents in SBEA. However, both the calcining temperature of 700 and 900 °C appeared to have no impact on the amount of LOI reduction being observed, leading to the conclusion that calcination temperature does not have significant effect on LOI mass reduction.

crystallinity of about 33 %. The crystalline components are predominantly SiO₂ in quartz form (Q) with XRD peaks reflected at 2θ of 20.88, 26.6 and 50.19 ° (Figure 6). Corundum, hematite and minor traces of brucite were also detected. This corresponds to the chemical composition

from XRF results in Table 3 as well as previous findings (Rahman *et al.*, 2019).

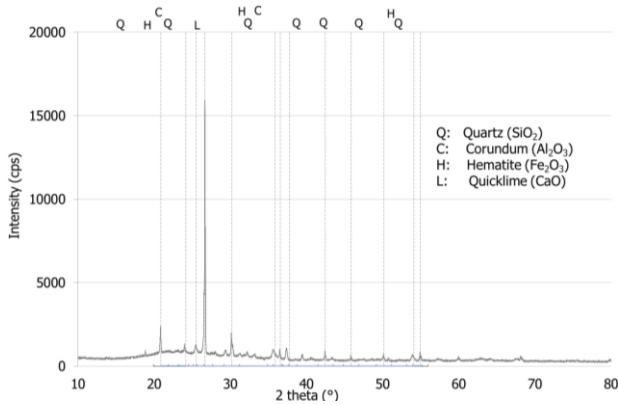


Figure 2. X-ray diffraction pattern for untreated SBEA (SU)

However, calcination of SBEA at 500, 700 and 900 °C, saw a massive reduction in the intensities of the quartz at 26.6 ° (Figure 7) across all three temperature ranges. This loss of intensity is typical when there is a loss of crystallinity in the quartz (Cao *et al.*, 2016). There was also a loss in the overall level of crystallisation (Table 5) observed in samples SC500 and SC700 associated with the loss in maximum peaks. This loss of peak and overall crystallinity should yield a more amorphous material state that is close to complete dehydroxylation.

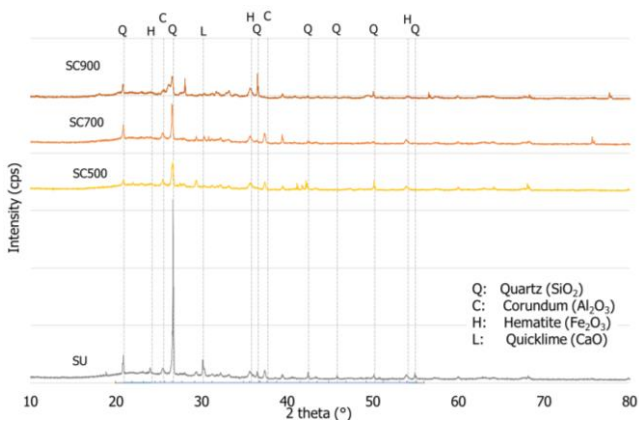


Figure 7. X-ray diffraction pattern for calcined SBEA at 500, 700 and 900 °C (SC500, SC700, SC900)

However, as calcination temperature increased to 900 °C (SC900), several new peaks started to appear, indicating overheating that resulted in the formation of crystallised mullite (Cao *et al.*, 2016) which is undesirable for strength formation. As a result, the overall level of crystallinity has

also increased to 36 %, which is higher than SBEA in its original, untreated form. The tremendous impact this has on the compressive strength of the mortar samples can be seen in section C of the Results and Discussion part of this paper.

Table 5. Degrees of crystallinity

Level of treatment	No. of peaks	Degree of crystallinity	Maximum peak height/intensity
SU	25	33 %	80,793
SC500	25	31 %	3,206
SC700	32	30 %	4,301
SC900	36	36 %	2,677

B. Pozzolan Reactivity

1. Strength Activity Index

The pozzolanic reactivity of SBEA was assessed using strength activity index (SAI) method. As presented in figure 8, untreated SBEA (SU) can be seen to exceed the target strength index of 75% by a fair margin of 31.8 %. Calcination was able to improve the strength index (SI) further by 40.1% (SC500) and 45.9 % (SC700). This was expected based on past research where pozzolans calcined at 700°C was found to experience a good increase in pozzolanic reactivity (Ilić *et al.*, 2016). On the other hand, SC900 achieved a lower SI of 116.2 % which is lower compared to SC700, despite its higher calcination temperature. This can be explained by earlier observations in the XRD pattern in section 2. Chemical Properties which showed that SC900 with its extremely high calcination temperature of 900 °C had caused re-crystallisation and caused formation of mullite which is detrimental to strength.

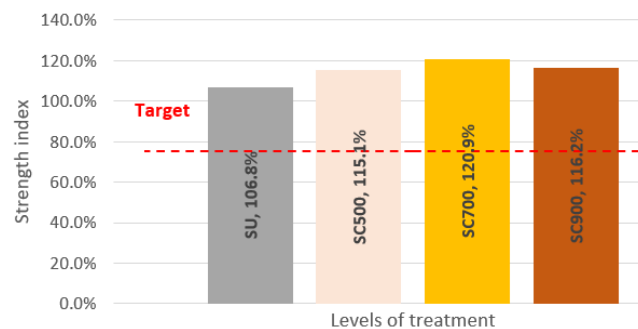


Figure 8. Strength activity index of samples

2. Residual CH Determination using TGA

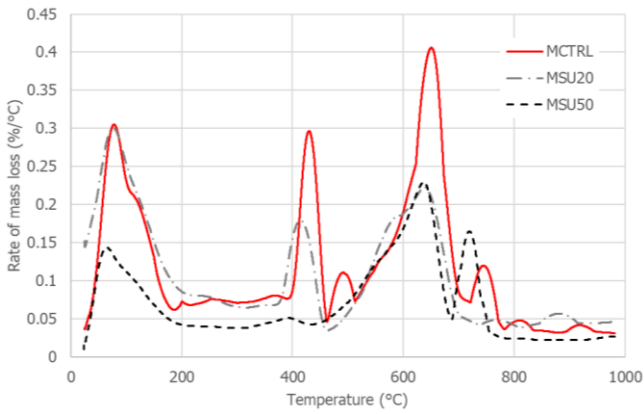


Figure 9. DTG graph showing decomposition of CH for samples MCTRL, MSU20 and MSU50

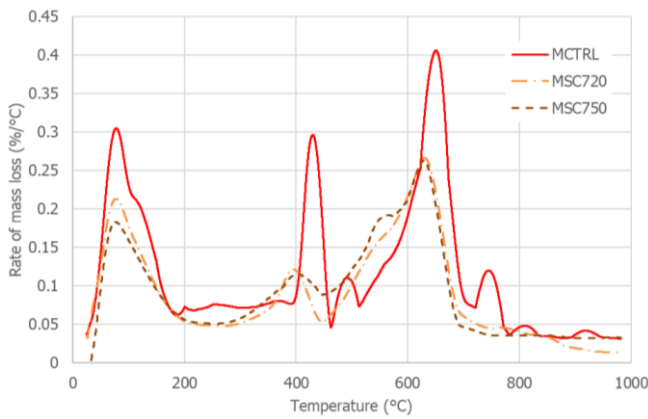


Figure 10. DTG graph showing decomposition of CH for samples MCTRL, MSC720 and MSC750

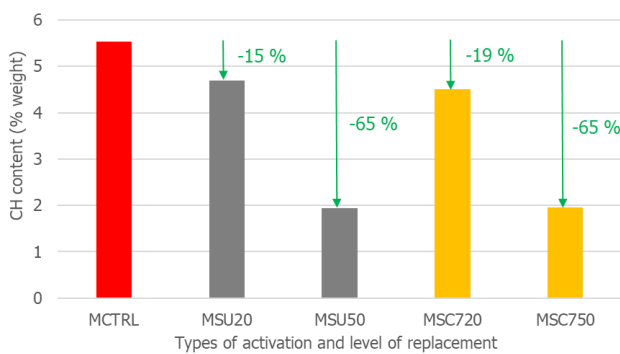


Figure 11. Residual CH content for all samples

C. Tests on Cement Mortar Samples

This section will evaluate the impact of both untreated (MSU) and calcined SBEA (MSC500, MSC700, MSC900) replacement in cement mortar in terms of water demand, as well as the compressive and flexural strengths.

1. Water Demand

While casting the cement mortar samples, it was expected that the use of natural pozzolan such as untreated SBEA (MSU) in cement mortar would increase the water demand as levels of replacement goes up (Figure 12). This was due to the smaller size and higher level of fineness of SBEA particles which required more water to hydrate.

However, this phenomenon could be reversed through the calcination of SBEA prior to its use in cement mortar as it could clearly be seen to have a positive impact in reducing the overall water demand across all testing ranges (Figure 12). The calcination temperature itself appeared to play little effect in affecting the water demand. The reduction achieved was as high as 11.5 % when compared against samples containing untreated SBEA and even at its lowest the amount of reduction observed was 2.2 % (Figure 13). This reduction in water demand can be attributed to reduction in LOI content such as water-absorbing carbon particles (Khankhaje *et al.*, 2016) as well as the reduction in specific surface due to the spherical nature of calcined particles (Ferreiro *et al.*, 2019). As the LOI content for both SC700 and SC900 calcined samples were similar, it is then as expected that the level of water demand would turn out to be similar as well.

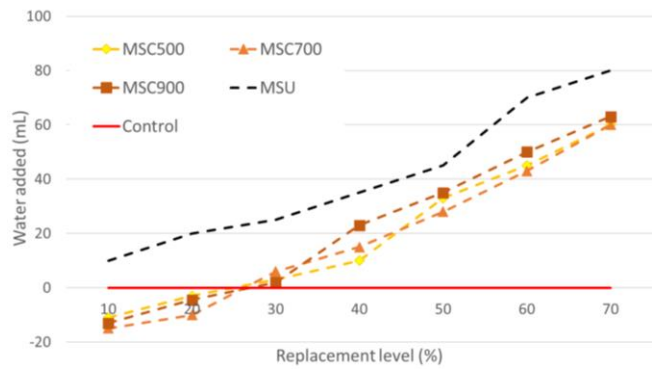


Figure 12. Water requirement of cement mortar samples

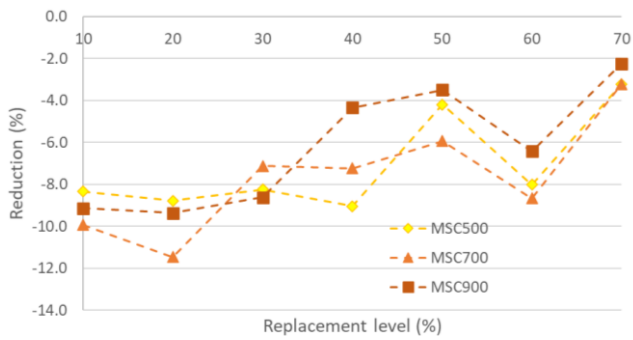


Figure 13. Percentage reduction of water demand for calcined SBEA

2. Compressive Strength

The early age (7-day) compressive strength trend of the mortar samples can be seen in Figure 14. As expected, use of pozzolans was detrimental to early age strength as we can see all samples; both untreated and calcined SBEA cement mortar under-performed against the control sample. This is due to a slower rate of reaction involving pozzolans as pozzolanic reaction required sufficient portlandite to be produced before it could commence. The worst performing batch was the MC900 series which recorded strengths of even less than that of samples with untreated SBEA (MSU). Meanwhile sample MSC500 performed very close to MSU and the only MSC700 managed to exceed the strengths of MSU samples by a significant amount at lower levels of replacement ($\leq 40\%$). The lack of strength development of mortar samples containing SBEA at early age was likely due to insufficient CH being produced to trigger pozzolanic reaction.

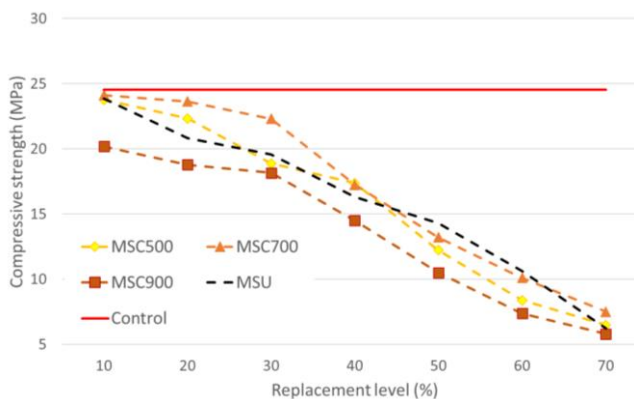


Figure 14. Compressive strength of cement mortar samples at 7 days

At 28 days, we can observe that most test samples were able to exceed the strengths of the control sample at levels of replacement under 30% (Figure 15). At this range, the worst performing batch is the MSU series with untreated SBEA. The calcined SBEA samples were all able to out-perform the MSU samples with peak strength achieved by MSC700 at 20% replacement level. Additionally MSC700 samples was also able to extend the allowable replacement level to 40% without comprising on strength. This behaviour exhibited by MSC700 can be attributed to higher amorphous content (Jaarsveld *et al.*, 2004) of the 700 °C calcined SBEA. Consequentially, at higher replacement levels past 40%, calcination hadn't managed to improve the strengths of the test sample with MSC900 being the worst performing. This is partly due to the re-crystallisation and the resulting loss of pozzolanic reactivity (Konan *et al.*, 2009) that occurred due to the excessive calcination temperature with the SC900 samples.

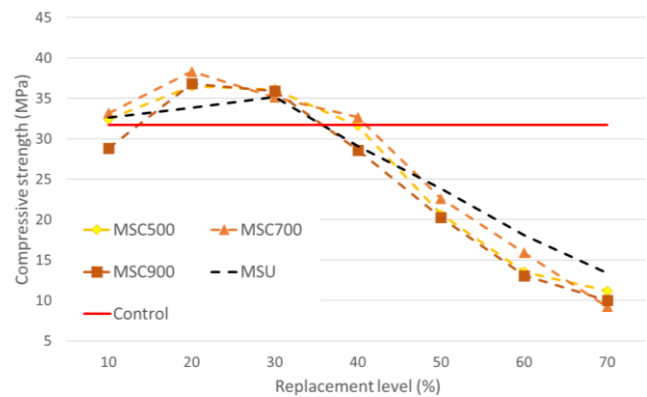


Figure 15. Compressive strength of cement mortar samples at 28 days

The long term compressive strength trend at 56 days were similar to the 28 day profile where MSU samples showed minor improvement over the control sample at low levels of replacement (Figure 16). Calcination of SBEA improved the strength of the test samples especially between replacement levels of 20 and 30%, and like the 28 day profile, the peak strength was achieved by MSC700 sample at 30% replacement. Again, we could also see that calcined samples (SC500 and SC700) were able to increase the levels of replacement past 40% as well as improving the overall strength against MSU samples at high levels of replacement ($> 40\%$). As opposed to portland cement mortars, cements

with higher amount of pozzolan will typically continue gaining strength at later ages due to the availability of pozzolan for reaction.

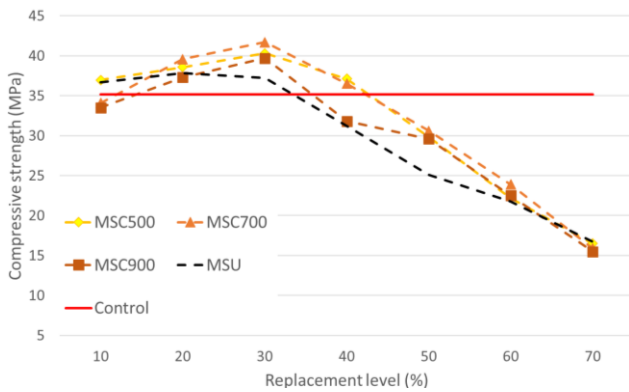


Figure 16. Compressive strength of cement mortar samples at 56 days

The observations from compressive strength test results appear to correlate well with data from past research that suggests pozzolanic reactivity and strength paste is highly influenced by the degree of amorphousness in a pozzolan (Walker & Pavía, 2011). It has also been noted in the past that pozzoanic reactivity increases linearly with calcination temperature up to 700, afterwhich the effect (Tironi *et al.*, 2012). This behaviour had been partially observed in this current experimental work but the linearity of the pozzolanic reactivity is somewhat less obvious.

3. Flexural Strength

As with its compressive strength counterpart, the flexural strength of MSU samples at 28-day out-performed the control sample at low levels of replacement (20 %) and under-performed at higher levels (50 %). However, it appeared that flexural strength benefitted very little from the calcination of SBEA as none of the samples could out-perform the samples with untreated SBEA (MSU) or the control (Figure 17). It would appear that in this instance, the flexural strength does not benefit from the calcination of SBEA, contrary to the observations recorded from the compressive strength testing.

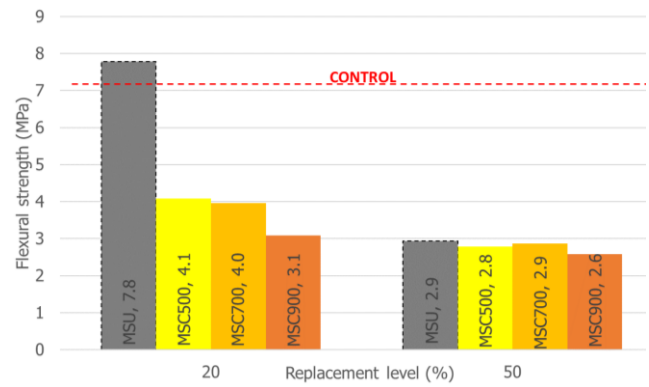


Figure 17. Flexural strength of cement mortar samples at 28 days

The accuracy of the flexural strengths obtained from testing were also validated against theoretical conversion formulas based on compressive strengths from past research. This was done by comparing the ratio of tested flexural strength/compressive strength, (f_{cu}/f_{cu}) against theoretical values estimated from compressive strength based on formulas by (*IS 456:2000 Plain Concrete and Reinforced - Code of Practice, 2000*), (*Guide for Design and Construction of Concrete Parking Lots Reported by ACI Committee 330, 2001*), (Croney & Croney, 1997) and (Kubica & Galman, 2022) as shown in Figure 18.

It can be seen that the theoretical values all exhibit similar trends with (Kubica & Galman, 2022)'s prediction forming the upper limit and IS 486/(Croney & Croney, 1997) prediction covering the lower end of the spectrum. The actual test results show that both MCTRL and MSU samples at 20 % replacement achieved higher strength ratios than theoretical ones but all the calcined samples as well as the MSU sample at 50 % both under-predicted against theoretical values. Also, at lower levels of replacement (0 % and 20 %), there does not appear to be good correlation between tested flexural strengths and theoretical predicted values at lower replacement levels but some semblance of correlation can be drawn at higher levels (50 %) between the tested strengths and those of Croney and Croney's.

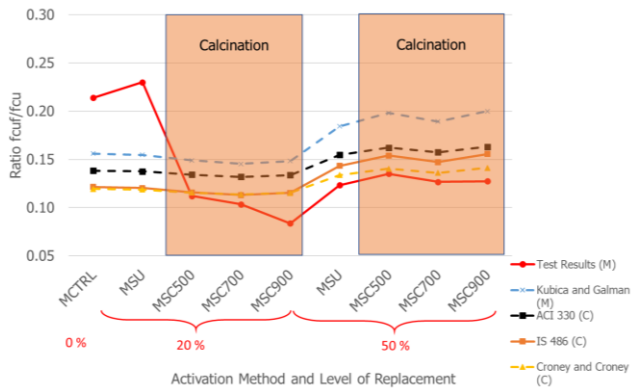


Figure 18. fcu/fcu ratio comparison between testing result and theoretical prediction

III. CONCLUSION

1. Untreated SBEA as supplied meets the chemical requirement of Class N natural pozzolan.
2. Calcination of SBEA can increase its amorphous content and at the same time reduce LOI but excessively high temperature will cause re-crystallisation (900 °C in this case).
3. Physically, calcination had little impact on changing the properties of SBEA except for its colour.
4. Untreated SBEA shows good pozzolanic reactivity in SAI tests and calcination improved this further but excessively high temperature (900 °C in this case) will negate this benefit.
5. Use of SBEA in cement mortar increases water demand but this could be reversed through calcination of SBEA.
6. Use of SBEA in cement mortar is detrimental to early age compressive strength (7-day) and calcination did little to improve this.
7. For 28 and 56-day compressive strength, SBEA replacement in cement mortar improved strength at replacement levels $\leq 30\%$ but calcination improved this further by allowing this replacement level to be extended to 40%.
8. Untreated SBEA appeared to have similar effect on flexural strength but calcination had the reverse effect, drastically reducing its flexural strength.
9. The ideal calcination temperature in this test appeared to be 700 °C as it allowed the maximum amount of amorphisation to occur as well as conferring the best improvement to compressive strength of cement mortars.
10. Calcination temperature of 900 °C is excessive as it causes re-crystallisation and loss of strength to cement mortar.

IV. ACKNOWLEDGEMENT

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