Determination of the Compressive Strength Properties of Alkali-activated Millet Husk Ash - Calcium Carbide Mortar

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Abstract
Alkali-activation of agro-industrial wastes as an alternative binder to Portland cement (PC) is receiving more consideration most notably in the developed nations due to issue of green-house gas (CO_2) emission from the production process of PC. This research focused on developing a suitable proportion combination of millet husk ash (MHA) – calcium carbide waste (CCW) and evaluate the fresh and hardened properties of alkali-activated MHA – CCW binder-based mortar sample. The chemical analysis of the supplementary cementitious materials via X-ray fluorescent (XRF) revealed MHA having 73.4% silica (SiO_2) content, while CCW primarily contains CaO (66.1%). Three combination proportions of MHA – CCW (40:60, 45:55 and 50:50) were activated with different molar concentrations of sodium hydroxide (NaOH): 5M, 10M, 15M and 20M respectively. The mortar was produced at 1:3 binder/sand (B/S) and 0.5 water/binder (W/B) examined for binding, hydration and strength development and water absorption at varied curing age (3, 7, 14, 28, and 56 days respectively) in accordance to BS EN 196-1: 2016. The MHA – CCW samples tested exhibit increasing performance for both properties examined with increasing NaOH concentration up to 15M but decreased performance at 20M for all combination proportions. At 56 days of curing the 45:55 MHA – CCW at 15M NaOH molarity possessing similar compressive strength and water absorption as the control.

Keywords: *Millet husk ash (MHA), Calcium carbide waste (CCW), Sodium hydroxide (NaOH), Alkali-activated binder, Compressive strength.*

INTRODUCTION
Cement is an important element in all types of construction for binder; and in recent years the cement market has been filled by one product, (Tsado *et al.*, 2014). In many countries, PC is expensive and this has severely limited the construction of affordable housing (Didel *et al.*, 2014). As a result, developing alternative binder to PC is an excellent option at much lower cost toward making a significant contribution in the provision of low-cost building materials leading to affordable shelters (Abdullahi *et al.*, 2013). Olawuyi *et al.* (2017) reported that PC based binder is one of the essential construction materials in the world produced through calcination of calcium carbonate ($CaCO_3$) to give calcium oxide and carbon dioxide (CO_2) emitted to the atmosphere as shown in equation (1.1).

$$CaCO_3 \xrightarrow{heat\ (>900\ C)} CaO + CO_2 \qquad (1.1)$$

$$CO_2 + SiO_2, + Al_2O_3 + Fe_2O_3 \xrightarrow{heat\ (>1450\ ^oC)} CaO, SiO_2, Al_2O_3\ \&\ Fe_2O_3 \qquad (1.2)$$

The basic components of a PC are calcium oxide (CaO), silica (SiO_2), alumina (Al_2O_3) and ferric (Fe_2O_3) with strength determinant being the SiO_2 which combined with CaO in the presence of water to form hydrated lime – $Ca(OH)_2$. This reaction leads to the formation of calcium silicate hydrate – CaO-SiO_2-H_2O (C – S – H), the end product of strength development in cement hydration process after water contact (Mehta & Monteiro, 2014). This article reports an attempt at alkali-activation of a pozzolan (as SiO_2 source) with a good CaO source as a complete replacement of PC towards proper binding and strength development properties in mortar and concrete.

Pozzolans are fine materials that contain silica and alumina which on their own have little or no binding property but when combined with lime in the presence of water, will set and hardened like cement (Abdulfatai *et al.*, 2013). The utilization of pozzolanic materials, for example, sawdust ash (SDA), rice husk ash (RHA), metakaolin (MK), fly ash (FA), silica fume (SF) and natural pozzolans as partial cement clinker replacement will help to minimize CO_2 emissions known to accompany cement production, (Damtoft *et al.*, 2008). Research trends on alternative binder had focused on the utilization of natural pozzolans i.e., volcanic ash (Hossain, 2003 & 2005; Hassan, 2006; Olawuyi, 2011; Hassan, 2016). Agricultural waste ashes such as RHA (Okpala, 1987; Chaowat, 2001; Abalaka and Okoli, 2013), SDA (Elinwa and Mahmood, 2002), corn-cob ash [CCA] (Raheem, 2010), millet husk ash [MHA] (Jimoh *et al.*, 2013) and palm kernel nut ash [PKNA] (Joshua *et al.*, 2015) amongst others having been used as a partial PC replacement in mortar or concrete. Olonade and Bello (2018) and Qureshi and Ghoshi (2013) on the other hand, worked on alkali-activated cocoa shell ash (CSA). According to Mehta and Monteiro (2014), pozzolanic reaction is the same pattern as that of PC using Tri-Calcium-Silicate (C_3S) with water (H) to give Calcium-Silicate-Hydrate (C-S-H) and Calcium Hydroxide (CH).
The (Portland- pozzolan) cement reactions are shown in equation (1.3) and (1.4) as follows:

$$C_3S + H \rightarrow C - S - H + CH \qquad (1.3)$$

$$\text{Pozzolan} + CH + H \rightarrow C\text{-}S\text{-}H \qquad (1.4)$$

Where C = CaO, S = SiO_2 and H = $(OH)^-$
The reaction in equation (1.3) is known to be fast and lime producing while the reaction in equation (1.4) is rather slow or latent depending on the properties of the pozzolanic material.

Alkali activation as defined by Martinez and Palomo (2001) is the chemical process where an amorphous structure is transformed into a skeletal structure that exhibits cementitious properties. The material containing reactive silica or alumina can be activated as shown in equation (1.5) and (1.6).

$$Na(OH)_2 + SiO_2 \rightarrow \text{N-S-H (Gel) as in geopolymer} \qquad (1.5)$$

$$Na(OH)_2 + Al_3O_2 \rightarrow \text{N-S-H (Gel) as in geopolymer} \qquad (1.6)$$

Where N-S-H (Gel) is Sodium-Silicate-Hydrate Gel.

Bakharev (2006) reported that concrete produced using alkali-activated fly ash with NaOH achieved a two-day compressive strength of 10 N/mm^2 while the 28days compressive strength was 60 N/mm^2. Bakharev (2006) also reported two days compressive strength of 2N/mm^2 and a 28-day compressive strength of 45 N/mm^2 from fly ash activated with sodium silicate (Na_2SiO_2).

EXPERIMENTAL PROCEDURE

Materials

The materials used for the study are MHA, CCW, NaOH, CEM II 42.5N (Dangote 3X cement) and fine aggregate. The MHA was obtained from Garatu village near Minna) in Bosso Local Government Area of Niger State, Nigeria. The husk was burnt in an open-air with locally fabricated incinerator presented earlier in Abalaka (2013). This material was ground to finer particles at Central Services Laboratory of National Cereals Research Institute (NCRI), Badeggi, near Bida, Niger State and sieved with a 75 µm sieve and the particles passing used as the MHA –SiO_2 sources for the study. CCW– an industrial waste of acetylene gas production was collected from the disposal area of a local automobile Welder's ("Panel Beater's") workshop in the mechanic village of Keteren-Gwari, Minna, Niger State. It was sun-dried for a day and calcinated in a furnace at a temperature of 700^o C to obtain its amorphous form and was used as the CaO source. The NaOH used was purchased from Panlac Chemical Laboratory, Minna.

Methods

The study involved determination of physical and chemical properties of the constituent materials, suitable proportion combination of MHA- CCW were assessed for strength development and water absorption properties of the alkali-activated binder-based mortar samples considered. PC based mortar samples of 1:3 (C/S) at 0.5 water/cement ratio (W/C) prescribed in BS EN 196-1:2016 served as control while for the alkali-activated binder-based mortar, varied proportion combinations of MHA-CCW with different concentration of NaOH were prepared and tested for the strength development and water absorption at requisite curing ages of 3, 7, 14, 28 and 56 days. The test procedures are further discussed sections 2.2.4 and 2.2.5.

Determination of Physical Properties of Fine aggregate, MHA and CCW

Particle size distribution test was conducted on the natural sand using the dry-sieve approach in accordance with BS EN 933-1 (1997) for proper classification of the sand sample. The particles passing the 1.18mm sieve but retained on the 75µm sieve was used for the study. The 75µm sieve was used as the limit value for sand used as presented in Figure 1 in consonance with BS EN 196-1 (2016) reference sand prescription for strength test on cement. The specific gravity of all materials used in this study was determined following the provisions of BS EN 1097 (2013). Also, the fineness test on the various combinations of MHA–CCW with varied molarities of NaOH and PC (CEM II 42.5N) were determined using the method as specified by BS EN 196-6 (2016) using the 53µm sieve.

Particle size distribution (PSD) and BET specific surface area of supplementary cementitious materials (SCMs) used was conducted with the use of Malvern Zetasizer Instrument at the Centre for Genetic Engineering and Bio-informatics Technology, Federal University of Technology, Minna, Niger State.

Determination of Chemical Composition of Cement, MHA and CCW

X-ray fluorescence (XRF) analysis for the oxide composition was conducted on the cementitious materials (MHA, CCW and PC) at National Geoscience Research Laboratory, Kaduna State.

Determination of Mixing Proportion of MHA and CCW

The suitable mix proportions of MHA and CCW was determined adopting the molar ratio concept taking cognizance of the SiO_2 and CaO contents of MHA and CCW respectively to arrive at 40:60; 45:55; 50:50 of MHA-CCW combination proportions. For the control mortar mix, CEM II, 42.5N was used at 1:3 C/S at 0.5 W/C. NaOH at different concentrations of 5, 10, 15, 20M was prepared for the study. The choice of NaOH concentrations was based on the findings from the Olonade and Bello, (2018) and Ammar *et al.*, (2013).

Determination of Compressive Strength of Mortar Sample

The compressive strength is a key property of mortar to which numerous of its characteristic are related (Neville, 2012). 50 mm cube mortar samples were cast and cured by water immersion for the requisite ages (3, 7, 14, 28 and 56 days) before testing for compressive strength. The mortar cubes were removed from the curing tank and put in the open air in the laboratory to surface dry, weighed and placed at the centre of hydraulic Digital Universal Testing Machine (DUTM - 20) for crushing force (P) determination in consonance with BS EN 196-1 (2016). The compressive strength was calculated using equation (2.1).

$$CS = \frac{P}{A} \tag{2.1}$$

Where;

CS = compressive strength in N/mm^2; P = maximum load at failure in N (Newton) and A = cross-sectional area, in mm^2.

Determination of Water Absorption of Mortar Sample

The water absorption test involved removing the mortar cubes were removed from the curing tank and allowed to surface-dry before placed in the electric oven to the oven-dry at 105 ^oC for 72 hours. The oven was then switched off, allowed to cool back to room temperature before removing the test samples and weight measurements taken and recorded as initial weight (w_1). The final weights were determined after immersing the mortar samples in the curing medium for 30 minutes, removed, towel-dried, re-weighed again and the value was recorded as (w_2). The water absorption of the mortar samples was then calculated using equation (2.2) under BS 1881-122, (2011)

$$Water\ Absorption = \frac{(w1 - W2) * 100}{w1} \tag{2.2}$$

RESULTS AND DISCUSSION

Characterization of the constituent Materials

Summary of the sieve analysis on the natural sand as presented in Onuche et al. (2019) is shown in Table1 and revealed the sand to be well-graded and of fine classification (C_c = 2.5, C_u = 1.0 and FM = 2.3) in line with Shetty (2004) specification. MHA was also reported as meeting the quality criteria of even dispersal, giving an average particle size of 180.23 nm and BET specific surface area value of 360.5 m^2/g. The data quality of the CCW sample was, however, observed to be poor and poly-disperse for distribution analysis. The CCW sample has an average particle size of 109.01 nm and BET specific surface area of 414.245 m^2/g.

Table 1: Summary of Sieve Analysis of Natural Sand

D10	D30	D60	Cc	Cu	FM
0.22	0.34	0.55	2.50	0.96	2.30

Specific Gravity of Constituent Materials

The specific gravity value of the constituent materials is presented in Table 2. The results indicate that the values fit well with the previous report in (Neville, (2012).

Table 2: Specific Gravity of Constituent Materials (kg/m^3)

Cement	MHA	CCW	NaOH	Sand
3.14	2.20	2.09	1.65	2.62

Chemical composition properties of MHA and CCW

The oxides composition of different cementitious materials was conducted at National Geoscience Laboratory, Kaduna. The MHA is a class N Pozzolan since the aggregation of the main oxides (SiO_2 + Al_2O + Fe_2O_3) gives 88.1% which is above the 70% minimum limit stipulated in ASTMC 618 (2012) standard as presented in Table 3.

***Table 3:** XRF Analysis for Oxide Composition of Cementitious Materials*

Samples	SiO_2	Al_2O_3	LOI	Fe_2O_3	MgO	CaO	K_2O	TiO	MnO	SrO	CuO	ZnO	BaO	SO_3	SiO_2+Al_2O+Fe_2O_3
MHA	72.4	14.2	2.0	1.5	1.0	6.4	0.2	0.0	0.5	0.0	0.0	0.2	0.1	0.1	88.1
CCW	5.5	1.8	28.2	0.3	0.1	66.1	0.1	0.1	0.0	0.0	0.0	0.0	0.1	0.2	7.6
PC	21.3	5.1	0.0	1.1	2.8	65.0	0.0	0.1	0.0	4.4	0.0	3.5	0.0	3.5	27.5

Also, SO_3 is below 4% and Loss on Ignition (LOI) is less than 10%. The CCW was observed to contain 66.1% CaO, a similar value to the CaO content (65.0%) of the PC and lower SiO_2 and Al_2O_3. The LOI of CCW is above the specified 10% maximum which shows that there might still be presence of impurities in the sample.

Density

The average density of the control and various combination proportions of activated MHA-CCW at 28 and 56 days respectively are presented in Figures 4a, 4b and 4c. The average density of alkali-activated MHA-CCW mortar samples and the control varies from 2099 kg/m^3 to 2182 kg/ m^3. It showed that the density increased as the curing age increases. Mortar samples with density above 2000 kg/ m^3 are considered as normal weight mortar/concrete according to ASTM C 140:2003. Thus, higher density recorded by mortar from these alkali-activated binders was at 15M (45:55 MHA-CCW) and thereby still falls under the normal weight mortar classification.

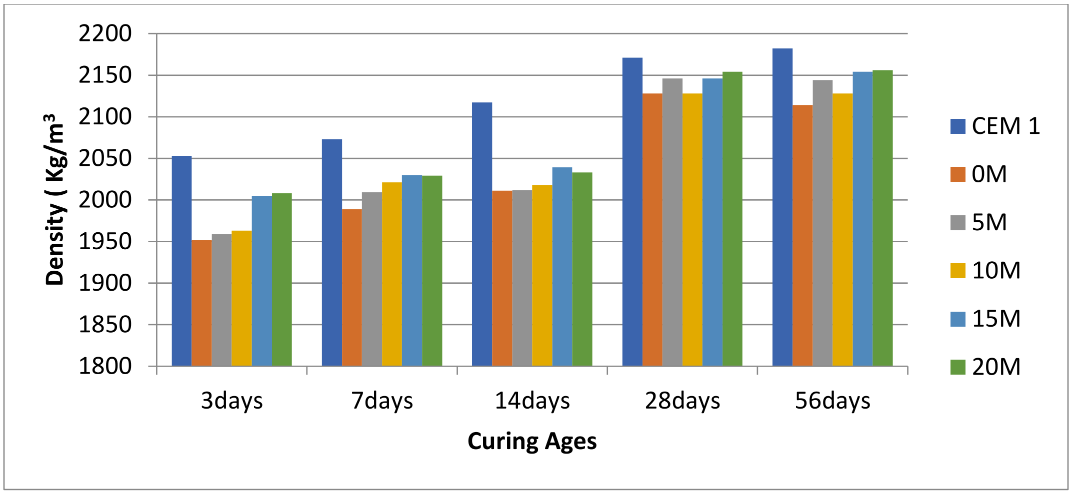

Figure 4a: Average density of 40:60 MHA/CCW mortar samples cured in water

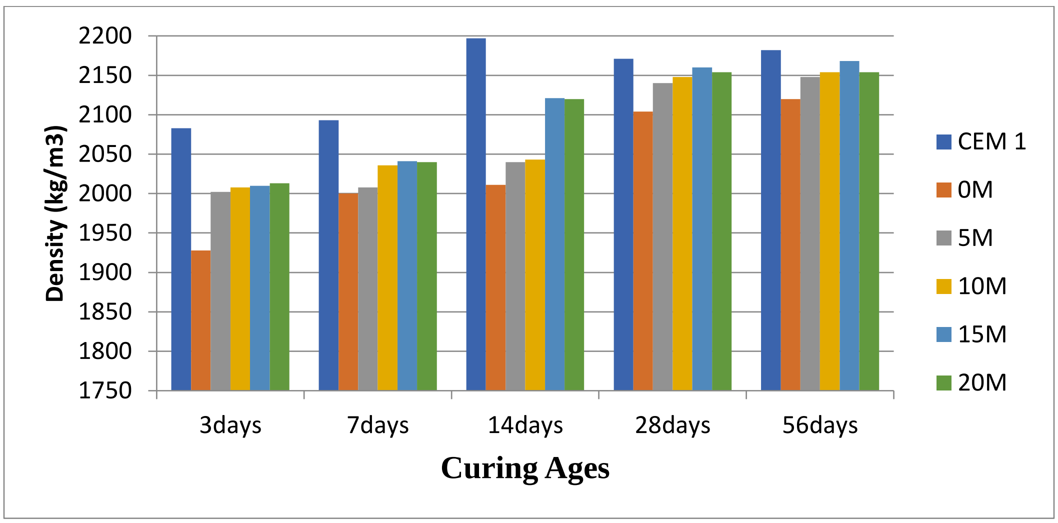

Figure 4b: Average density of 45:55 MHA/CCW mortar samples cured in water

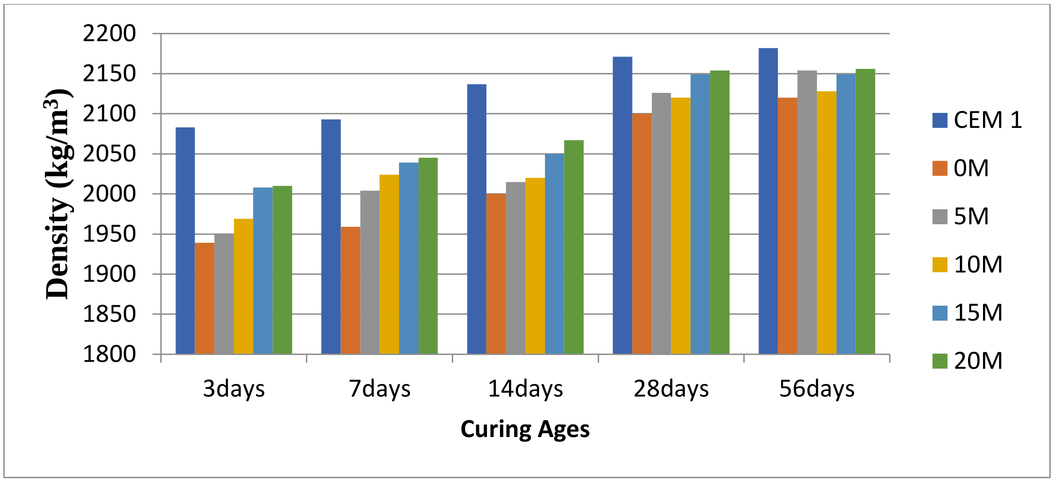

Figure 4c: Average density of 50/50 MHA/CCW mortar samples cured in water

Compressive strength of Alkali Activated MHA/CCW (AAMHA-CCW) Mortar Samples

The compressive strength of alkali-activated mortar at varied molar concentrations of NaOH solution is shown in Table 4. At the early age of 3 days, the 45:55 (MHA/CCW) activated with 15M of NaOH produced the highest strength of 6.92 N/mm^2 (i.e. 35% of its 28^{th}-day strength – CS_{28}) which is higher than the stipulated minimum strength of $3N/mm^2$ for load-bearing sandcrete blocks as postulated by the Nigeria Industrial Standard (2004).

Table 4: Compressive Strength of Alkali-Activated MHA-CCW Mortar

Specimens		NaOH (M)	Compressive Strength (N/mm^2)					CS_{28} Factor				
			3days	7days	14days	28days	56days	3days	7days	14days	28days	56days
PC		0	10.24	16.02	20.26	26.29	28.04	0.39	0.61	0.77	1.00	1.07
MHA/CCW	40/60	0	1.80	1.96	3.33	7.44	7.92	0.24	0.26	0.45	1.00	1.06
		5	2.68	3.38	5.35	9.76	11.48	0.27	0.35	0.55	1.00	1.18
		10	4.59	6.98	9.71	13.84	15.28	0.33	0.50	0.70	1.00	1.10
		15	6.52	9.00	15.33	18.45	22.66	0.35	0.49	0.83	1.00	1.23
		20	4.00	5.85	10.17	16.02	18.52	0.25	0.37	0.63	1.00	1.16
	45/55	0	1.96	2.08	3.84	7.68	8.20	0.26	0.27	0.50	1.00	1.07
		5	2.80	3.91	5.44	10.28	12.20	0.27	0.38	0.53	1.00	1.19
		10	4.77	7.78	10.19	14.69	16.44	0.32	0.53	0.69	1.00	1.12
		15	**6.92**	**10.72**	**15.96**	**19.77**	**24.52**	**0.35**	**0.54**	**0.81**	**1.00**	**1.24**
		20	4.72	6.12	10.56	16.45	20.64	0.29	0.37	0.64	1.00	1.25
	50/50	0	1.84	2.00	3.76	7.23	8.04	0.25	0.28	0.52	1.00	1.11
		5	2.72	3.87	5.41	10.00	11.56	0.50	0.27	0.39	0.54	1.00
		10	4.65	7.53	9.95	14.36	15.88	0.32	0.52	0.69	1.00	1.11
		15	6.80	9.95	15.48	19.32	23.24	0.35	0.52	0.80	1.00	1.20
		20	4.20	5.96	10.21	16.25	19.88	0.26	0.37	0.63	1.00	1.22

As the curing age of AAMHA-CCW increased, it was observed that the rate of strength development increased with increase in the concentration of NaOH from 5M to 15M but at 20M, slight decrease in strength was observed. This trend implies that the maximum strength recorded for AAMHA-CCW on the 56th day was at 45:55 proportion combination activated with 15M of NaOH, the similar value was observed for the 50:50 samples at 15M NaOH. This result conforms to the findings by Ramujee and Potharaju (2014) which reported that the compressive strength of alkali-activated binder increases as the molar concentration of NaOH increased up to 15M. This concentration of NaOH resulted in good bonding between the aggregate and the mortar paste until the maximum concentration for activation is achieved.

The strength of the AAMHA-CCW mortar also increased as the curing ages increased for all the proportions combinations of MHA-CCW. The 7- and 14-days strength for 45:55 MHA-CCW activated with 15M NaOH were 10.72 N/mm^2 and 15.96 N/mm^2 i.e.67% and 79% respectively of the PC-based sample at the same age. The 28-days strength for the 45:55 MHA-CCW, 15M NaOH sample showed higher strength gain of 29% as against 23% strength gain of the PC-based mortar. Further curing of AAMHA-CCW mortar till 56days resulted in an additional 24% strength gain over the 28th day as compared to the 7% increase of the control. The trend is the same for all the AAMHA-CCW mortar studied up to 15M NaOH concentrations. This study considers the 15M NaOH activation of 45:55 MHA-CCW as the indicated proportion for good strength.

CONCLUSION AND RECOMMENDATIONS

In this study, agro-industrial waste materials (MHA and CCW) were activated at varied molarities of NaOH for performance assessment at utilization as an alternative binder in mortar as compared to PC based mortar. The chemical analysis reveals MHA as class N pozzolan of high SiO_2 content (72.4%) while CCW is good CaO source showing percentage concentration of 66.1% similar to the control. The PSD analysis and BET specific surface showed the MHA particles distribution to be of good quality and dispersed uniformly while the CCW particles are too poly-disperse for distribution analysis due to its fluorescence and sediment particle nature. The compressive strength results of AAMHA-CCW at 15M NaOH showed the best combination proportions and of similar characteristics as the PC based mortar. At 56 days of curing, AAMHA-CCW mortar cube samples revealed 15M of NaOH and 45:55 of MHA-CCW possessing compressive strength value near that of control.

Based on the finding of this study, the following are recommended:

i. Future studies on the product of hydration should be conducted using scanning electron microscopy (SEM) and X- ray diffraction (XRD) analyses.
ii. Influence of further heat treatment on the CCW sample and the effect on the performance of the alkali-activated binder should be investigated.
iii. There were potential that the AAMHA-CCW could be further explored using different optimization approach to produce higher strength for structural use, like normal strength mortar.

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