PROJECT NO. RU-14

ANNUAL REPORT COMPREHENSIVE RESEARCH ON RICE
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PROJECT TITLE: The effects of post-pyrolysis treatments on the properties of rice straw for use in cement-based materials

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OBJECTIVES AND EXPERIMENTS CONDUCTED, BY LOCATION, TO ACCOMPLISH OBJECTIVES:

All experiments and/or analyses done to accomplish these objectives were performed on the University of California, Davis campus. To complete some of the proposed work, ash samples have been sent to an analytical facility off-campus to determine oxide composition and morphology in a timely manner.

In this work, we focus on post-combustion processing to improve the performance of rice straw ashes and allow higher cement replacement rates. Physiochemical and material properties of ash and ash blended in cement-based materials are determined through a combination of analytical and physical testing. Two means for improving consistency of ashes as mineral admixtures are explored: (1) the first is to assess the effects of grinding the ashes, (2) the second method for improving consistency is to blend the RSAs with another mineral additive (in this case, ground granulated blast furnace slag). Milling or co-blending RSA were considered for its potential improve the performance of RSA and allow for higher cement replacement levels [18]. The results of this work will inform which post-combustion treatment methods are most effective at obtaining desirable properties from RSA.

2. Materials and Methods

2.1. Materials and binder constituents

Mortar and paste specimens were produced to experimentally evaluate rice straw ash in cement-based materials. Type II/V Portland cement (PC) was purchased (BASALITE Concrete Products, Dixon, California) for all mixtures. Mortars mixtures used silica-sand acquired from the Esparto quarry in Esparto, California. Ground blast furnace slag (GBFS) was acquired from Lehigh-Hanson Cement (Stockton, California). Rice straw ash was produced by West Biofuels, with the goal to mimic the combustion process of biomass for energy productions. Rice straw was locally sourced from Windmill Feed (Woodland, California) and milled in a hammermill with a 1.25-inch (32 mm) diameter round-hole screen. Rice straw was first torrefied at 250°C for 40 minutes, to prevent premature ignition, and then oxidized for 6 hours at 500°C. After combustion RSA was used untreated (U-RSA), milled (M-RSA), or blended with GBFS. For M-RSA, RSA was prepared in a planetary ball mill using 3 mm Zirconium grinding medium in a Zirconium grinding jar to pass a #200 sieve (74um).

The chemical composition of the binder materials used in this work were evaluated for likely oxide composition, trace elements, loss on ignition (LOI), and amorphous content by Activation Labs (Ontario, Canada). Likely oxide composition and trace elements were determined using inductively coupled plasma optical emissions spectroscopy (ICP-OES). Trace elements, a very small proportion of the total materials, are evaluated to understand how co-blending will change the prevenance of these elements compare to PC or GBFS. The relative amorphous content was determined by X-ray diffraction using an internally mixed coronium standard to semi-quantitively determine the phase abundance. In this method, the relatively amount of identified phases are crystalline and the remaining unidentified phases are considered to be amorphous in structure. LOI was determined by measuring mass loss of a 2g sample in a furnace at 1000°C for 2 hours.

2.2. Compressive strength

The compressive strength of cement-based materials is a key metric used in design. To be an effective cement replacement, the RSA needs to provide similar performance despite variations in agricultural practice and combustion conditions. To determine if any of the treatments improved consistency of RSA performance across ash type, 7-day and 28-day compressive strengths of RSA-cement mortar cube specimens were performed. Mortar mixtures were prepared with a water-to-binder (w/b) ratio of 0.59 and a cementitious material-to-sand ratio of 2.5. Five sets of mortar mixtures were produced: (1) a control mixture with 100% PC as the cementitious material; (2) a control mixture with 70% PC and 30% GBFS; (3) a mixture with 85% PC and 15% U-RSA; (4) a mixture with 85% PC and 15% M-RSA; and (5) a ternary-blended cementitious material with 30% GBFS, 10% U-RSA, and 60% PC (using the same replacement ratios as Gursel *et al.* [18] tested for RHA-PC-Fly Ash mixtures). The mortar mixture proportions are provided in **Error! Reference source not found.**.

Table 1. Mortar mixture composition and proportions for compressive strength testing

Mixture Names	w/b	Constituent Proportions (kg per cubic meter of mortar) *				
		PC	Sand	RHA	Slag	Water
100% PC (CTRL)	0.59	528	1321	0	0	312
GBFS - 30%	0.59	370	1321	0	158	312
U-RSA	0.59	444	1306	78	0	308
M-RSA	0.59	444	1306	78	0	308
30%GBFS - 10%URSA	0.59	315	1311	52	157	309

*PC – Portland cement, RSA – Rice Straw Ash, GBFS – Ground blast furnace slag, Water – Distilled Water, w/b – water-to-binder ratio

Mortar cube specimens measuring approximately 51 mm X 51 mm (2 inches X 2 inches) were made following the mixing procedures in ASTM C109 [19]. Specimens were consolidated with a vibrating table, for approximately 30 seconds, prior to curing at <90% RH and 25°C. After one day, specimens were demolded and then returned to the curing chamber to cure at <90% RH and 25°C until being removed at 7-day or 28-day for compressive strength testing. As described in ASTM C109 [19], mortar cubes were loaded in compression between two platens using a CT-950 load frame, under force control, until failure occurred. The maximum load achieved was used to calculate the compressive strength of each specimen. Five specimens, at each age, were used to determine the average compressive strength and standard deviation of the compressive strength for each mortar mixture.

2.3. Isothermal calorimetry

Isothermal calorimetry can be used to assess heat of hydration, which informs our understanding of cement behavior during curing. The isothermal calorimetry experiments were performed in a TAM air isothermal calorimeter (TA instruments, New Castle, Delaware) with the internal temperature of 25°C. Isothermal calorimetry was performed by in-situ mixing paste samples in 20 mL glass ampoules. Paste mixtures with an approximate mass of 5 grams were produced with a w/b ratio of 0.59. Powder was proportioned into the glass ampoules and the distilled water was measured using syringes. The tips of syringes were dried and then dipped in silicon to prevent premature mixing of the powder and liquid components. Reference samples of similar thermal capacity as paste were made from inert sand. Before hydration and mixing the paste, the powder filled ampoules and syringes with distilled water were equilibrated in the calorimeter. After equilibrating, a baseline reading was take using the software package. For each sample, the liquid portion was injected over approximately 5 seconds, the time of injection was noted, and the sample was then mixed for ~1.5 minutes. After data collection finished, the software package was used to automatically correct the data using the baseline heat flow and the noted liquid addition time was used to mark the correct reaction start time.

SUMMARY OF 2023 RESEARCH (major accomplishments), BY OBJECTIVE:

1. Chemical composition of cementitious materials

Little difference was measured in amorphous content between the U-RSA (93.3% amorphous) and the M-RSA (93.9% amorphous) ashes. This high amorphous content is related to the comparatively low combustion temperature (500°C) of the ash. Typically, high amorphous content relates to higher reactivity of the material as an SCM, which can be beneficial to strength development of cement-based materials.

As with the amorphous content, the likely oxide analysis shows little change between the untreated and milled conditions for the rice straw ash **Table 2**. The greatest mass different was between the SiO_2 (~2%); however, both values are within the range of 72.2% – 95.6% reported in literature [9]. For RSA, all the oxide composition except for Na_2O fall within commonly reported ranges [9]. The maximum Na_2O value for rice straw reported in the Phyllis 2 database (across 15 samples) was 1.85% [20], and this value was reported from an untreated rice straw that was also collected in Northern California [21]. The sample tested here differs from this result by 0.15%, which can likely be attributed to various agrarian factors, such as differences in weather, changes in soil conditions, and other cultivation decisions. The Portland cement and GBFS chemical composition display CaO as the predominate oxide with a relatively large amount of SiO_2, as well. For the cement, the amount of Al_2O_3, Fe_2O_3, and MgO are all within the allowable limits prescribed in ASTM C150 [22].

Table 2. Likely oxides of binder constituents

Oxide	Detection Limit	U-RSA	M-RSA	PC	GBFS
SiO_2 (%)	0.01	76.37%	78.55%	22.37%	32.21%
Al_2O_3 (%)	0.01	0.42%	0.35%	3.71%	15.50%
Fe_2O_3 (%)	0.01	0.54%	0.42%	3.15%	0.41%
MnO (%)	0.005	0.32%	0.27%	0.08%	0.24%
MgO (%)	0.01	2.18%	1.80%	0.96%	8.89%
CaO (%)	0.01	2.21%	1.82%	68.77%	39.89%
Na_2O (%)	0.01	4.25%	4.09%	0.26%	0.38%
K_2O (%)	0.01	12.52%	11.73%	0.40%	0.48%
TiO_2 (%)	0.001	0.02%	0.02%	0.18%	1.96%
$P2O_5$ (%)	0.01	1.16%	0.95%	0.11%	0.03%

The U-RSA and M-RSA has very similar trace element profiles to each other, except for Zirconium (Zr) content. This additional Zr is likely from milling in a ballmill with Zr medium in a Zr lined jar. However, this increased Zr concentration (172 ppm) remains much lower than that of the slag SCM (311 ppm). Notably, all other values are below the trace element concentrations in the Portland cement, suggesting the presence of these trace elements would not lead to deleterious effects in a cement-based composite. LOI for the ashes was 9.69% (untreated) and 11.3% (milled). The LOI of slag is negative, which indicates a mass increase from oxidation during LOI testing (which could occur from oxidation of sulfur species [23]).

Table 3. Trace elements in binder constituents

Trace element	Detection Limit	U-RSA	M-RSA	PC	GBFS
Ba (ppm)	2	93	71	260	615
Sr (ppm)	2	106	82	1133	707
Y (ppm)	1	3	12	20	67
Sc (ppm)	1	-	-	3	28
Zr (ppm)	2	22	172	57	311
Be (ppm)	1	-	-	1	9
V (ppm)	5	8	6	165	66
LOI (%)	0.01	9.69	11.3	2.23	-1.36

'-' denotes measurement below detection limit

2. Compressive strength

The average compressive strengths of each mixture at 7 days and 28 days are shown in **Figure 1** as well as presented in **Table 4** with standard deviations. At 7 days, the U-RSA compressive strength was approximately 2% higher than the mortars made with M-RSA (**Figure 1a**). Compared to the average 7-day compressive strength of the CTRL mortar, the untreated RSA 7-day strength was ~18% lower and the milled RSA 7-day strength was ~20% lower. For comparison, the 30% GBFS control mixture exhibited also a 18% lower compressive strength compared to the 100% PC control. At 28 days, the mortar mixtures with SCMs experienced greater rates of strength development than the 100% PC control mixture, but the mixtures with RSA remained on average lower strength than the 100% PC control mixture. The M-RSA compressive strength was 8% higher than the U-RSA mixtures (**Figure 1b**), but compared to the 100% PC control mixture, the U-RSA mixture had 11% lower strength, and the M-RSA had ~4% lower strength. The 30%-GBFS control mixture had ~1% higher compressive strength than the 100% PC control mixture (i.e., a negligible difference). This delayed strength gain may be attributed to a delayed pozzolanic reaction during binder hydration and development. Notably, while at 7 days, the ternary cement blend (30% GBFS, 10% U-RSA, and 60% PC) had 14% lower strength than the U-RSA mixture; at 28 days, the ternary blend performed similarly to the U-RSA mixture. While some compressive strength was lost compared to the 100% PC and 30% GBFS control mixtures (~13%), these findings suggest additional material optimization and appropriate application selection could enable a low-clinker-content ternary cement blend with acceptable mechanical performance.

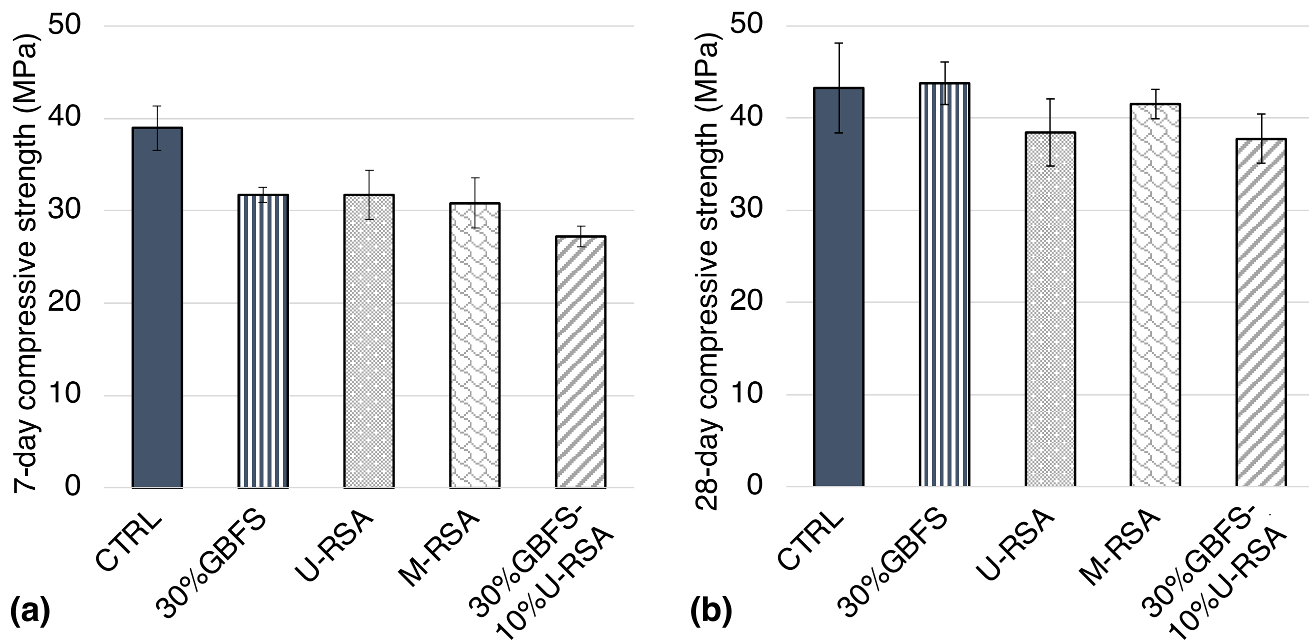

Figure 1. Average compressive strength of Rice straw ash (RSA) mixtures at **(a)** 7-days and **(b)** 28-days. Bars show the standard deviation.

The compressive strengths of the RSA mixtures are shown in **Table 4** along with the compressive strength ranges of analogous rice hull ash (RHA) mixtures made with one of four different RHAs from the same industrial combustion facility (from prior work). The ternary mixture (30% GBFS, 10% U-RSA, and 60% PC) had a higher 7-day average then the ternary blended RHA mixture. For all other mixtures, the RSA average compressive strength falls within the same range as the analogous RHA mixtures.

Table 4. Table showing the average compressive strength of the RSA mixtures with the standard deviation given in parenthesis. The range of compressive strengths measured in analogous mixtures made with one of 4 different RHA (part of prior work) is provided for comparison.

Mixtures	RSA compressive strength, MPa		Compressive strength range for comparable RHA mixes, MPa			
	7d	28d	7d Min	7d Max	28d Min	28d Max
CTRL	39.0 (2.4)	43.3 (4.9)	--	--	--	--
30%GBFS	31.7 (0.8)	43.8 (2.3)	--	--	--	--
U-RSA	31.7 (2.7)	38.4 (3.7)	20.4	33.6	29.6	44.2
M-RSA	30.9 (2.7)	41.5 (1.6)	25.0	34.2	36.1	50.1
30%GBFS - 10%U-RSA	27.2 (1.2)	37.7 (2.6)	20.1	26.0	33.0	44.6

Value in parenthesis is the standard deviation. Values are rounded to a tenth of a one MPa.

A one-way analysis of variance (ANOVA) of 7-day compressive strengths indicates that the difference in mean compressive strengths is statistically significant ($F(2.86) = 14.66$, $p = 9.53 \times 10^{-6}$). Pairwise comparisons of the mixtures using the Tukey HSD post-hoc test indicates that the mean variations between the CTRL mixture and all other mixtures are statistically significant ($Q = 6.51$; 6.51; 7.28; 10.54 for mixtures 30%GBFS, U-RSA, M-RSA, 30%GBFS-10%U-RSA, respectively, and $Q_{critical} = 4.23$ for confidence interval, $\alpha = 0.05$). However, differences in the mean 7-day compressive strengths between the non-CTRL mixtures with each other was not found to be statistically significant (all Q values $< Q_{critical}$). The reduced mechanical strength at 7-days for mixtures with Portland cement replacement led to meaningful variations in the average compressive strength. This suggest that the RSA and GBFS had less beneficial contributions to the compressive strength compared to the Portland cement at this early age. Comparing the mean 28-day compressive strengths using a one-way ANOVA indicates that group means do vary in a statistically significant way, albeit with values that just meet the threshold: $F(2.86) = 2.90$, $p = 0.047$ (confidence interval, $\alpha = 0.05$). However, the Tukey HSD post-hoc test does not show statistically significant variation between specific groups when the mixture are compared pairwise (Q ranges from 0.32 – 3.73 for $Q_{critical} = 4.23$). This change from the 7-day strengths suggests that the RSA and GBFS materials may have a delayed contribution to the strength development of the mixture with little benefit to early age (7-day) strength. However, after 28-days; the mechanical performance between each of other mixtures and the CTRL mixture, when compared pairwise, does not vary in a significant way.

3. Isothermal calorimetry

The effect of treatment on the exothermic hydration reactions in cement paste was measured with isothermal calorimetry. Representative heat flow measurements are shown in **Figure 2**. When results are normalized to cementitious material, **Figure 2a**, shows that mixtures with 30% GBFS (i.e., 30%GBFS - 10%URSA and 30% GBFS control mixtures) had notable lower heat flow in the first 24 hours compared to mixtures with larger fractions of PC.

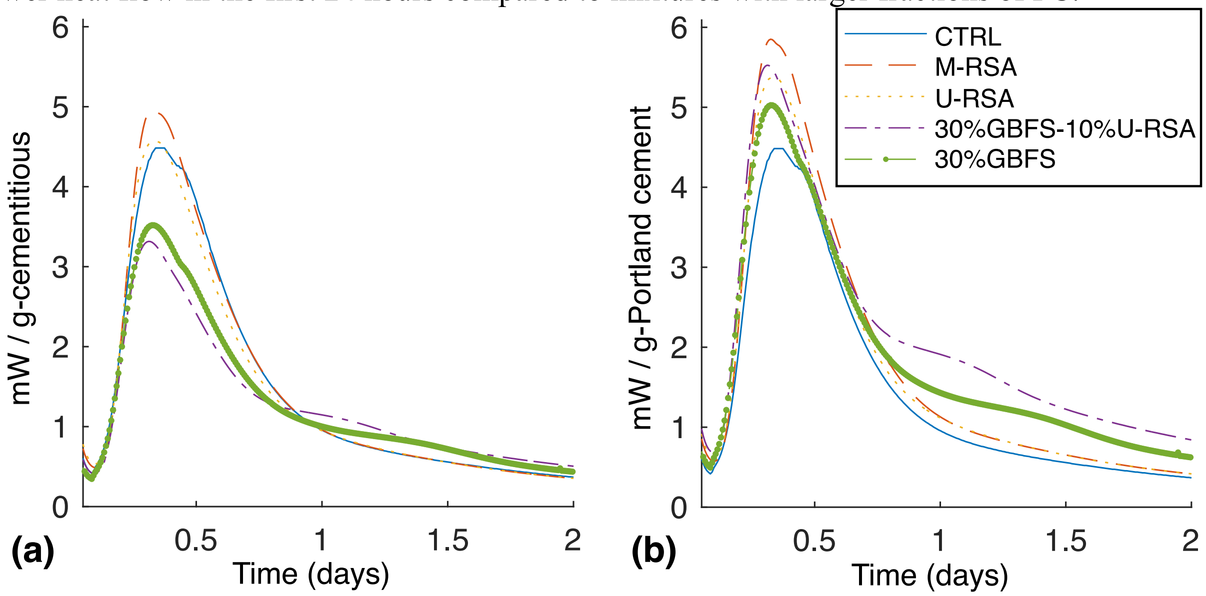

Figure 2. Heat flow of paste mixtures over 48 hours. (a) is normalized to the mass of cementitious material (i.e., the sum of Portland cement, Rice straw ash, and slag), (b) normalized to the mass of Portland cement only

In the same figure, the U-RSA had a very similar heat release as the 100% PC control specimen, and the M-RSA demonstrated the largest heat flow. The M-RSA blend also has the largest heat flow at the main hydration peak when normalized to the PC content (**Figure 2b**). These results suggest milling increases the reactivity of the RSA-cement blend. The GBFS blends also produce higher primary peaks when normalized to the PC, again suggesting increased reactivity from the SCM. Notably, after 1 day (generally regarded as the primary hydration region), the GBFS-containing mixtures have a higher heat flow (**Figure 2**), suggesting that the slag has a more delayed contribution to hydration and could contribute more to strength at later ages. This observation is further supported by the cumulative heat development of the pastes (shown in **Figure 3**). As noted in the heat flow, the slag mixtures have a lower heat development than the PC-RSA only mixtures. However, the GBFS paste show a more rapid heat development at later ages. Thus, all mixtures achieved a similar cumulative heat at 7 days of approximately 300 J / g-cementitious material.

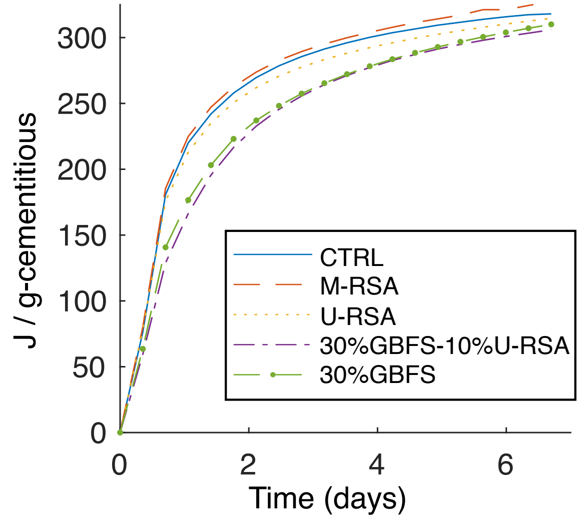

Figure 3. Cumulative heat development of paste mixtures over 7-day measurements, normalized to the mass of cementitious material (i.e., the sum of Portland cement, Rice straw ash, and slag)

PUBLICATIONS OR REPORTS:

A report will be compiled summarizing the work done and the findings from this research. It is also anticipated that at least one peer-reviewed publication will result from this research.

CONCISE GENERAL SUMMARY OF CURRENT YEAR'S RESULTS:

In this work, rice straw ash (RSA) is investigated as a supplementary cementitious material (SCM) to partially replace Portland cement (PC). RSA untreated, milled, and blended-with-blast furnace slag are each considered. Through evaluations of ash compositions, mortar compressive strengths, and the heat of hydration of cementitious pastes, this work provides and initial evaluation of the suitability of RSA as a reactive SCM. Notably, this work shows:

- At 28 days, ternary blended 30% GBFS and 10% U-RSA mixtures achieved similar compressive strengths as the 15% U-RSA mixtures.
- Milled and untreated rice straw ashes achieved similar compressive strengths.
- Milled RSA showed a slight increase in average strength at 28 day, but no improvement at 7 days compared to untreated RSA.
- Mixtures with GBFS (i.e., 30% or more PC replacement) showed a delayed reaction with lower initial heat of hydration (0-24h), but total heat reached similar levels as the CTRL at ~7 days.

- All RSA mixtures performed similarly to analogous rice hull ash mixtures tested in earlier studies.

Importantly, this study shows that rice straw ashes can perform similarly to rice hull ashes as SCMs in cement-based materials. With similar efficacy, the use of RSA could greatly expand viable rice biomass-derived SCMs for use in concrete materials as more straw is produced than hulls on a mass basis during rice cultivation. Future studies should consider the effects of rice straw ashes on durability and longevity of cement-based materials. Additionally, work that advances rice-straw based combustion systems to concurrently drive energy generation and biomass SCM production could greatly improve the environmental benefits of utilizing this agricultural residue from farms.

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