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RESEARCH ARTICLE

RICE HUSK EFFECTS ON EARLY PORTLAND CEMENT HYDRATION

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Abstract. *This study evaluates the influence of dry rice husk (RH) on the early-age hydration kinetics of Portland cement. Temperature evolution monitoring was used to analyze the effects of RH content (5–15% by mass of cement) and pre-conditioning regimes (2 h water soaking and hot-water treatment at 100 °C) on maximum temperature rise (ΔT_{max}), time to peak hydration (t_{max}), reaction-rate indicator (R), suppression coefficient (K_s), and retardation index (R_i) relative to a control cement paste. The results show that raw RH significantly modifies hydration behavior by reducing heat evolution and delaying the acceleration stage. Increasing RH content progressively decreased ΔT_{max} and prolonged t_{max}. At 15% RH after thermal pre-conditioning, ΔT_{max} decreased by approximately 77% compared to the control, while the retardation index reached R_i = 5.99, indicating a substantial delay in peak hydration. Scanning electron microscopy (SEM) revealed that increasing RH content leads to a more heterogeneous and porous microstructure of the hardened cement paste, with hydration products forming around rice husk particles. These microstructural features correspond with the observed suppression of hydration kinetics. The results indicate that untreated rice husk acts as an active kinetic modifier rather than an inert filler in cement systems due to its high water absorption capacity and interaction with the pore solution chemistry.*

Keywords: *portland cement, hydration kinetics, rice husk, temperature evolution, hydration, water absorption, lignocellulosic materials*

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ҒЫЛЫМИ МАҚАЛА

КҮРІШ ҚАУЫЗДАРЫНЫҢ ПОРТЛАНДЦЕМЕНТТІҢ ЕРТЕ ГИДРАТАЦИЯСЫНА ӘСЕРІ

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Аңдатпа. *Бұл жұмыста құрғақ күріш қауыздарының (RH) портландцементтің ерте кезеңдегі гидратация кинетикасына әсері сандық тұрғыда бағаланды. RH мөлшерінің (цемент массасының 5–15%) және алдын ала өңдеу режимдерінің (2 сағат бойы суға жібіту және 100 °C температурадағы ыстық сумен өңдеу) максималды температураның көтерілуіне (ΔT_{max}), гидратация шыңының уақытына (tmax), реакция жылдамдығының көрсеткішіне (R), бәсеңдету коэффициентіне (K_s) және кідіру индексіне (R_i) әсері температура эволюциясын бақылау әдісі арқылы зерттелді. Зерттеу нәтижелері шикі күріш қауызын енгізу гидратация процесін айтарлықтай өзгертетінін көрсетті: жылу бөліну қарқындылығы төмендеп, үдеу кезеңі баяулайды. RH мөлшерінің артуымен ΔT_{max} мәні біртіндеп төмендеп, tmax ұлғаяды. 15% RH және термиялық алдын ала өңдеу жағдайында ΔT_{max} бақылау құрамымен салыстырғанда шамамен 77%-ға азайды, ал кідіру индексі R_i = 5,99 мәніне жетті. Сканирлеуші электрондық микроскопия (SEM) нәтижелері RH мөлшерінің артуы цемент тасындағы микроструктураның біркелкі еместігін және кеуектілігінің жоғарылауын көрсететінін анықтады. Сонымен қатар гидратация өнімдері күріш қауыздарының бөлшектерінің айналасында түзілетіні байқалды. Алынған нәтижелер өңделмеген күріш қауыздарының цемент жүйесінде инертті толтырғыш емес, гидратация кинетикасын өзгертетін белсенді компонент екенін көрсетеді.*

Түйін сөздер: *портландцемент, гидратация кинетикасы, күріш қауыздары, температуралық эволюция, гидратация, су сіңіру қабілеті, лигноцеллюлозалық материалдар*

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НАУЧНАЯ СТАТЬЯ

ВЛИЯНИЕ РИСОВОЙ ШЕЛУХИ НА РАННЮЮ ГИДРАТАЦИЮ ПОРТЛАНДЦЕМЕНТА

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Аннотация. *В данной работе проведена количественная оценка влияния сухой рисовой шелухи (RH) на кинетику гидратации портландцемента на ранних стадиях твердения. Для анализа влияния содержания RH (5–15% от массы цемента) и режимов предварительной обработки (замачивание в воде в течение 2 ч и обработка горячей водой при 100 °C) на максимальное повышение температуры (ΔT_{max}), время достижения пика гидратации (t_{max}), показатель скорости реакции (R), коэффициент подавления (K_s) и индекс замедления (R_i) применялся метод мониторинга температурной эволюции. Результаты показали, что добавление сырой рисовой шелухи существенно изменяет характер гидратации, снижая интенсивность тепловыделения и замедляя стадию ускорения. С увеличением содержания RH наблюдалось постепенное уменьшение ΔT_{max} и увеличение tmax. При содержании 15% RH после термической предварительной обработки ΔT_{max} уменьшалось примерно на 77% по сравнению с контрольным составом, а индекс замедления достигал R_i = 5,99. Исследования методом сканирующей электронной микроскопии (SEM) показали, что увеличение содержания RH приводит к формированию более неоднородной и пористой микроструктуры цементного камня, при этом продукты гидратации образуются вокруг частиц рисовой шелухи. Полученные результаты свидетельствуют о том, что необработанная рисовая шелуха выступает не как инертный легкий наполнитель, а как активный модификатор кинетики гидратации цемента.*

Ключевые слова: *портландцемент, кинетика гидратации, рисовая шелуха, температурная эволюция, гидратация, водопоглощение, лигноцеллюлозные материалы*

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CONFLICT OF INTEREST

The authors state that there is no conflict of interest.

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During the preparation of this manuscript, the authors used artificial intelligence tools (ChatGPT) solely for editorial assistance, such as improving phrasing and checking grammar, spelling, and punctuation. All ideas, interpretations, and conclusions are the responsibility of the authors, who take full accountability for the content of the article.

АЛҒЫС / ҚАРЖЫЛАНДЫРУ КӨЗІ

Зерттеу Қазақстан Республикасы Ғылым және жоғары білім министрлігі Ғылым комитетінің AP26101925 «Өсімдік шикізатының қалдықтары негізінде 3D басып шығаруға арналған құрылымдық-жылу оқшаулағыш бетон» гранттық қаржыландыру шеңберінде жүргізілді.

МҮДДЕЛЕР ҚАҚТЫҒЫСЫ

Авторлар мүдделер қақтығысы жоқ деп мәлімдейді.

Мақаланы дайындау барысында авторлар жасанды интеллект құралдарын (ChatGPT) тек редакциялық көмек мақсатында пайдаланды: тұжырымдарды жетілдіру, грамматикалық, орфографиялық және тыныс белгілеріндегі қателерді тексеру үшін. Барлық идеялар, интерпретациялар мен қорытындылар авторларға тиесілі, және олар мақаланың мазмұнына толық жауапты.

БЛАГОДАРНОСТИ/ИСТОЧНИК ФИНАНСИРОВАНИЯ

Исследование проводилось в рамках грантового финансирования Комитета науки Министерства науки и высшего образования Республики Казахстан AP26101925 «Конструкционно-теплоизоляционный бетон для 3D печати на основе отходов растительного сырья».

КОНФЛИКТ ИНТЕРЕСОВ

Авторы заявляют, что конфликта интересов нет.

При подготовке рукописи авторы использовали инструменты искусственного интеллекта (ChatGPT) исключительно для редакторской поддержки: корректировки формулировок, проверки грамматических, орфографических и пунктуационных ошибок. Все идеи, интерпретации и выводы принадлежат авторам, которые несут полную ответственность за содержание статьи.

1 INTRODUCTION

The development of sustainable cementitious materials has become an important direction in modern construction materials science. The increasing demand for environmentally friendly building materials has stimulated intensive research on the utilization of industrial and agricultural by-products in cement-based systems. The incorporation of such materials contributes to reducing environmental impact, improving resource efficiency, and decreasing the consumption of natural raw materials traditionally used in cement production (**Park et al., 2016; Wang et al., 2024**).

Agricultural residues are considered promising alternative materials for use in cementitious composites due to their availability and unique physicochemical properties. Among them, rice husk has attracted considerable attention because of its high silica content and potential influence on the hydration processes of cement-based binders. Large quantities of rice husk are generated annually as a by-product of rice milling, and its utilization in construction materials represents an effective approach for waste valorization and sustainable material development (**Xu et al., 2015; Marangu et al., 2020**).

Previous studies have mainly focused on the use of rice husk ash (RHA) obtained after controlled combustion of rice husk. Due to its high amorphous silica content and pozzolanic activity, RHA can react with calcium hydroxide released during cement hydration, leading to the formation of additional calcium silicate hydrate (C–S–H) phases and improvement of the microstructure of hardened cement paste (**Park et al., 2016; Wang et al., 2024**). As a result, the incorporation of RHA has been associated with enhanced mechanical properties, durability, and reduced permeability of cement-based materials.

However, while the pozzolanic properties of rice husk ash have been extensively investigated, significantly less attention has been paid to the influence of raw rice husk particles on the hydration behavior of Portland cement. Unlike ash obtained after thermal treatment, raw rice husk contains organic components and lignocellulosic structures that may interact with the cement pore solution and influence hydration processes through physical and chemical mechanisms. Understanding the interaction between raw rice husk and hydrating Portland cement is important for evaluating its potential use as a functional component in lightweight cementitious composites. Therefore, further investigation is required to clarify how the presence of untreated rice husk affects hydration kinetics and the formation of hydration products in cement-based systems.

Rice husk (RH) has been widely investigated as a supplementary cementitious material due to its high amorphous silica content and pozzolanic reactivity. When incorporated into cement systems, RH can react with calcium hydroxide released during cement hydration, forming additional calcium silicate hydrate (C–S–H) phases and contributing to microstructural densification of hardened cement paste (**Xu et al., 2015; Marangu et al., 2020**). As a result, the use of RH in cementitious composites has been associated with improvements in strength development and durability.

Several studies have investigated the influence of rice husk ash on the hydration behavior of cement-based materials. Experimental research indicates that the presence of rise husk ash (RHA) can modify the hydration process by affecting the dissolution of clinker phases and the formation of hydration products (**Park et al., 2016**). In particular, the incorporation of finely divided RHA particles may accelerate secondary reactions with calcium hydroxide, which leads to additional formation of C–S–H phases and refinement of the microstructure of the cement matrix (**Wang et al., 2024**).

The hydration kinetics of cementitious systems containing rice husk ash have also been analyzed using calorimetric techniques. Previous studies demonstrate that RHA can significantly influence the heat evolution during early hydration stages, altering both the induction period and the rate of heat release associated with the formation of hydration products (**Liu et al., 2020; Liu et al., 2025**). These changes in heat evolution are associated with both physical filler effects and chemical pozzolanic reactions occurring in the cement system.

Microstructural investigations using scanning electron microscopy (SEM) have also provided valuable information on the interaction between RHA and the cement matrix. SEM observations have shown that the addition of rice husk ash affects the morphology and distribution of hydration products, leading to a more refined and compact microstructure of hardened cement paste (**Sivakumar & Ravibaskar, 2009**). Similar findings were reported in later studies where the presence of RHA contributed to the formation of dense C–S–H structures and reduction of capillary porosity in cement-based materials (**Wang et al., 2020; Wang et al., 2024**).

In addition to studies on rice husk ash, several researchers have investigated the influence of mineral and ash-containing additives on cement hydration and structural development of cementitious binders. These studies demonstrate that the incorporation of mineral additives can significantly modify hydration reactions, microstructural evolution, and the resulting properties of cement-based materials (**Saginov et al., 2025; Takirova et al., 2025; Maikonov et al., 2025**).

Despite extensive research on rice husk, relatively limited attention has been given to the influence of raw rice husk particles on the hydration behavior of Portland cement. Therefore, the present study aims to provide a quantitative assessment of the modification of the hydration kinetics induced by raw rice husk. Using temperature evolution monitoring, the influence of rice husk content (5–15% by mass of cement) and pre-conditioning regime on exothermic response (ΔT_{max}), time to peak hydration (t_{max}), and derived kinetic indices is evaluated relative to a pure cement paste control. The study seeks to clarify the combined physical and chemical mechanisms governing hydration suppression in cement systems incorporating untreated lignocellulosic particles.

2 MATERIALS AND METHODS

Ordinary Portland cement CEM I 42.5N according to EN 197-1 was used as a mineral binder to eliminate the influence of additional cementitious materials and isolate the effect of raw rice husk on the hydration kinetics. The cement was stored in sealed conditions at laboratory temperature (22 ± 2 °C) prior to testing to prevent moisture uptake. The visual appearance of the cement powder is presented in **Figure 1c**.

Raw rice husk (RH), obtained as an agricultural by-product from rice milling (Kyzylorda region), was used as a lignocellulosic component without grinding or chemical treatment. The husk particles exhibit an elongated geometry, low bulk density, and fibrous surface morphology (**Figure 1a, b**), which may influence the retention of water and the interaction with the chemistry of the pore solution.

Figure 1 – Materials used in the study: (a) raw rice husk (general view); (b) raw rice husk particles (magnified view); (c) portland cement [author's material].

Tap water was used that met laboratory standards for cement testing. The temperature of the mixing water was maintained at 22.9 ± 0.5 °C.

A three-component system (cement, rice husk, water) was investigated. The experimental matrix evaluated the rice husk content (5–15% by mass of cement) and pre-conditioning regime. The reference control mixture (C0) consisted of 200 g cement and 70 g water (W/C = 0.35). Rice husk was incorporated at 5% (10 g), 10% (20 g), and 15% (30 g). The added water mass was kept constant for all compositions to ensure comparability of hydration behavior.

The Water-preconditioned husk (RH-W) was soaked for 2 h at 20–25 °C. Water absorption, % was calculated using the equation:

$$W = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100\% \tag{1}$$

where m_{wet} – mass of materials in wet state, g; m_{dry} – mass of materials in dry state, g.

The measured water absorption was 133%.

Thermal pre-conditioning (RH-T) consisted of immersion in water at 100 °C for 45 min followed by cooling to laboratory temperature.

The cement and rice husk were mixed dry prior to adding water. The addition of water defined time zero (t = 0). The fresh paste was transferred to a thermally insulated container for temperature monitoring.

Temperature evolution was recorded using a multi-channel acquisition system at 1-minute intervals. The maximum temperature rise (ΔT_{max}), the time to peak (t_{max}), the reaction-rate indicator (R), the suppression coefficient (K_s) and the retardation index (R_i) were calculated relative to the control paste coefficient $K_s = R_i/R_{C0}$, and the retardation index $R_i = t_{max,i} / t_{max,C0}$. The hydration characteristics of the fresh RH paste were measured using an exothermic profile according to the ALCOA methodology (**Almatis, 1999**) and evaluated according to (**Okino et al., 2004**). The evolution of exothermic temperature during early hydration was monitored using the experimental setup shown in **Figure 2**.

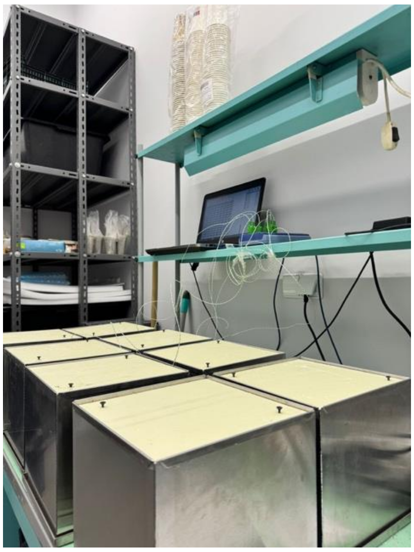

Figure 2 – Experimental setup used to monitor the evolution of temperature during cement hydration (author's material)

Scanning electron microscopy (SEM) was used to investigate the microstructure of the hardened samples. A small fragment of the specimen (approximately 10 mm) obtained after the compressive strength test was used for analysis. The fragment was cleaned from dust by air blowing and placed on a 25 mm metal sample holder. The prepared sample was then introduced into the SEM chamber for microstructural observation at different magnifications. The analysis procedure followed the general methodology described by **Kuldeyev et al. (2025)**.

3 RESULTS AND DISCUSSION

Figure 3 and **Figure 4** present the temperature evolution curves for mixtures containing 5%, 10%, and 15% RH under RH-W and RH-T regimes, respectively. For both regimes, increasing RH content led to a lower peak temperature and a systematic shift of the main peak toward longer times, indicating simultaneous suppression (reduced intensity) and retardation (delayed peak) of hydration. Notably, the 15% RH-T mixture exhibits a pronounced delay and a markedly reduced peak, reflecting a strong inhibition regime.

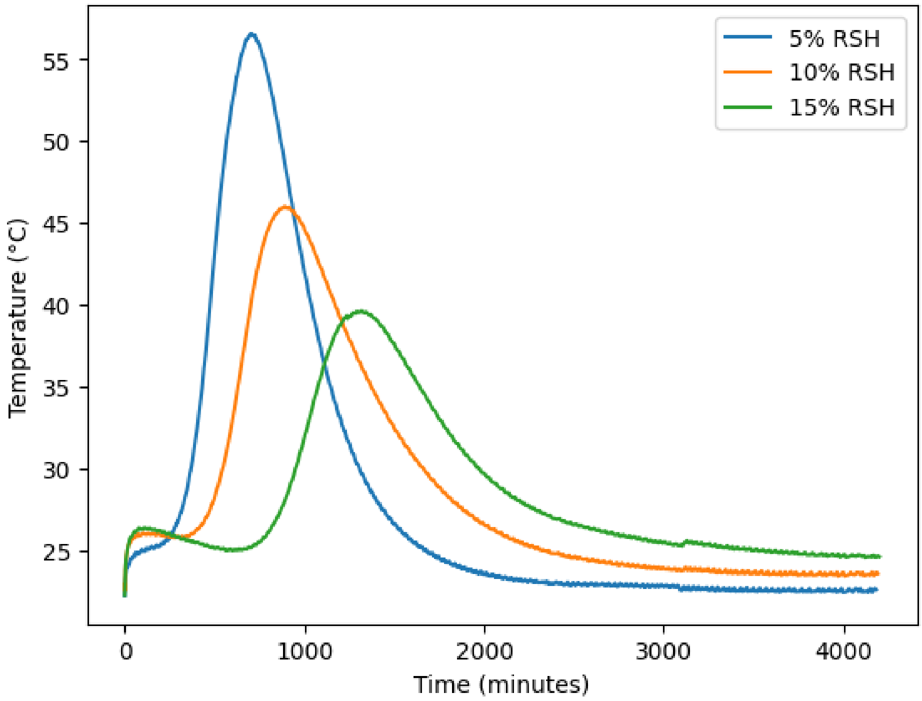

Figure 3 – Evolution of the temperature for cement pastes incorporating rice husk after 2 h of water pre-conditioning (RH-W) (author's material)

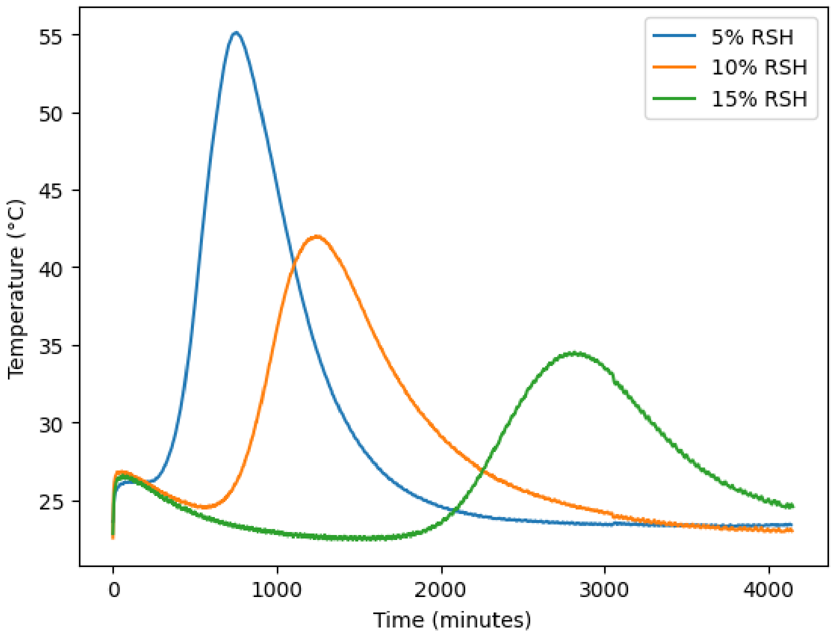

Figure 4 – Evolution of the temperature for cement pastes incorporating rice husk after hot-water pre-conditioning (RH-T) (author's material)

The control cement paste (C0) showed ΔT_{max} = 50.62 °C and t_{max} = 469.6 min, corresponding to R_{C0} = 0.1078 °C/min. Findings in **Table 1** demonstrate the kinetic parameters for all mixtures and provide a dimensionless framework referenced to C0.

Table 1
Hydration kinetic parameters derived from temperature evolution (author's material)

Mixture	ΔT_{max}, (°C)	t_{max}, (min)	R, (°C/min)	K_s	R_i
CO	50.62	469.6	0.1078	1.00	1.00
RH5-W	34.36	704	0.0488	0.45	1.50
RH10-W	23.12	892	0.0259	0.24	1.90
RH15-W	17.38	1319	0.0132	0.12	2.81
RH5-T	31.55	756	0.0417	0.39	1.61
RH10-T	19.42	1241	0.0156	0.15	2.64
RH15-T	11.72	2814	0.00416	0.039	5.99

For RH-W mixtures, ΔT_{max} decreased from 34.36 °C (5%) to 17.38 °C (15%), while t_{max} increased from 704 min to 1319 min. The suppression coefficient decreased from K_s = 0.45 to 0.12, indicating substantial reduction of hydration intensity even under moderate pre-conditioning. Thermal pre-conditioning (RH-T) intensified the effect at higher RH contents: ΔT_{max} decreased to 11.72 °C and tmax increased to 2814 min for 15% RH-T. The corresponding K_s = 0.039 indicates that the reaction-rate indicator dropped to ~4% of the control, while R_i = 5.99 shows an almost sixfold delay of peak hydration. The nonlinear nature of the response suggests a transition from moderate kinetic modification at 5% RH to a strongly inhibited regime at 15%, particularly after thermal conditioning.

The observed behaviour is consistent with coupled physical and chemical controls. Physical water redistribution: RH exhibits high water absorption (133%), retaining water within its porous structure and reducing the amount of free water available for clinker dissolution. This delays the buildup of ionic supersaturation required for C–S–H nucleation, extending the induction period and shifting tmax. Chemical inhibition: soluble organic extractives present in raw lignocellulosic materials may complex Ca^{2+} and interfere with hydrate nucleation and growth. The sharp decrease in R and K_s at high RH-T dosage suggests that chemical inhibition becomes dominant when thermal pre-conditioning likely increases the mobility/release of soluble compounds. Together, these mechanisms explain the simultaneous reduction of ΔT_{max} and strong delay of t_{max} observed in **Figures 3–4**.

The microstructure of cement paste modified with raw rice husk (RH) was investigated using scanning electron microscopy to evaluate the morphology of hydration products.

As shown in **Figure 5a**, the sample containing 5% RH exhibits a relatively dense microstructure with typical hydration products of Portland cement, mainly gel-like calcium silicate hydrate (C–S–H) phases and partially hydrated cement grains. Such microstructural features are characteristic of hydrated cement systems containing silica-rich additives (**Park et al., 2016**).

In the sample with 10% RH (**Figure 5b**), the microstructure becomes more heterogeneous, with hydration products forming clusters around RH particles. This indicates that the presence of rice husk influences the spatial distribution of hydration phases within the cement matrix.

For the sample containing 15% RH (**Figure 5c**), a more porous structure with visible voids is observed. The increased RH content reduces the compactness of the matrix, which is consistent with the changes observed in the heat evolution curves during hydration. Similar relationships between microstructural development and hydration behavior in cement systems containing rice husk-derived materials have been reported in previous studies (**Xu et al., 2015**).

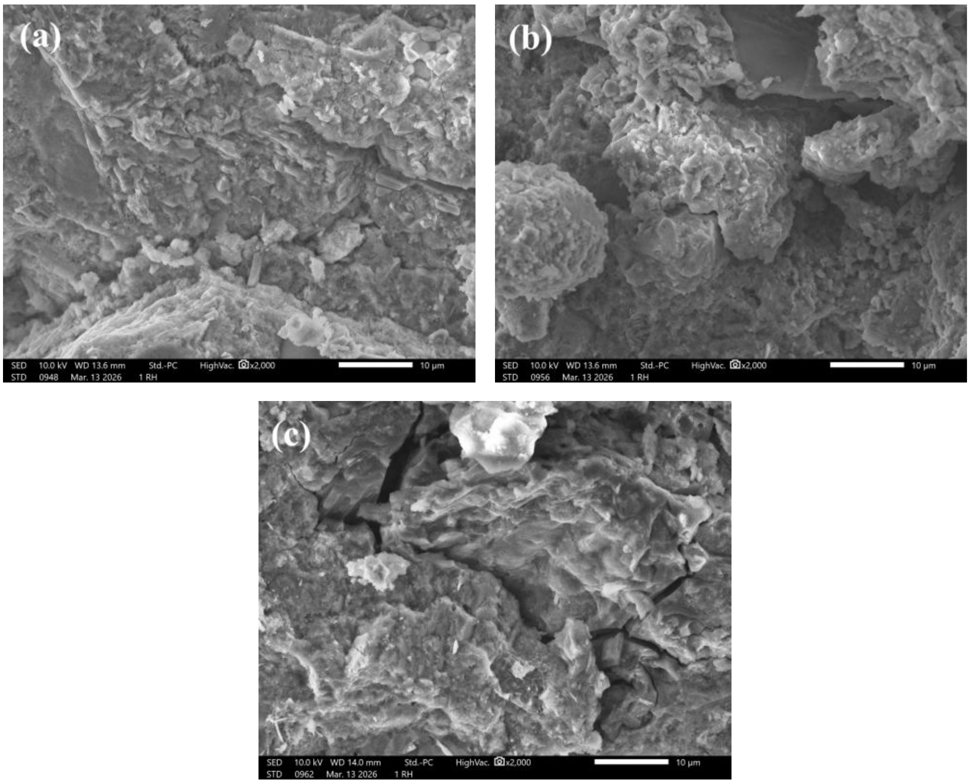

Figure 5 – SEM micrographs of hydrated cement paste modified with raw rice husk: (a) 5% RH; (b) 10% RH; (c) 15% RH. Magnification ×2000 (author's material)

From a material design perspective, the results indicate that raw RH cannot be treated as an inert lightweight filler in cement systems. At 10–15% RH, substantial hydration retardation is expected, potentially affecting setting and early-age strength. If high RH contents are targeted for lightweight/insulating applications, compatibility measures (optimized pre-treatment, binder selection, or chemical activation) may be required to mitigate excessive retardation.

4 CONCLUSIONS

This study quantitatively evaluated the influence of the content of raw rice husk (RH) and the pre-conditioning regime on the hydration kinetics of Portland cement using a temperature evolution analysis.

The results show that raw RH significantly modifies early-age hydration behaviour. Compared to control cement paste (ΔT_{max} = 50.62 °C; t_{max} = 469.6 min), the incorporation of RH led to simultaneous suppression of the intensity of heat evolution and delay of the acceleration stage.

Key findings include:

1. Systematic reduction in hydration intensity. The maximum temperature rise (ΔT_{max}) progressively decreased with increasing RH content. At 15% RH under thermal pre-conditioning, ΔT_{max} was reduced by approximately 77% relative to the control.

2. Pronounced delay of peak hydration. The time to peak temperature (t_{max}) increased significantly with RH content. The highest retardation index (R_I = 5.99) was observed for 15% RH-T, corresponding to nearly sixfold extension of the induction period.

3. Nonlinear suppression behaviour. The reaction-rate indicator (R) and suppression coefficient (K_s) revealed a nonlinear response to an increase in RH dosage. Although moderate modification occurred at 5% RH, a transition to strong kinetic inhibition was observed at ≥15%, particularly after thermal pre-conditioning.

4. Coupled physical and chemical mechanisms. The hydration modification is attributed to the combined effects of:

– internal water redistribution due to high RH absorption capacity (133%), reducing effective free water availability;

– chemical interaction of soluble organic extractives with pore solution chemistry, potentially interfering with Ca^{2+} availability and hydrate nucleation.

5. Microstructural observations. SEM analysis confirmed that the incorporation of raw rice husk influences the microstructural development of cement paste. At lower RH contents (5%), the cement matrix remained relatively dense with typical hydration products. Increasing RH dosage led to a more heterogeneous and porous structure, indicating reduced compactness of the hydration products. These observations are consistent with the calorimetric results showing suppression and delay of hydration reactions.

6. Practical implications. Raw rice husk cannot be treated as an inert lightweight filler in cement systems. At higher doses (≥10%), significant retardation must be expected, which may affect setting time and early strength development. Optimized pre-conditioning strategies or binder adjustments may be required to mitigate excessive kinetic suppression.

In general, the study confirms that untreated lignocellulosic additives act as active kinetic modifiers in cement systems. The derived dimensionless indices (R, K_s, R_i) provide a quantitative framework for assessing hydration suppression in bio-modified cementitious materials.

Further investigations combining calorimetric analysis and microstructural characterisation are recommended to isolate the relative contributions of physical water redistribution and chemical inhibition mechanisms.

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