

Article

Mechanical and Environmental Performance of Chemical Pretreated Incineration Bottom Ash as a Supplementary Cementitious Material

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Abstract

Municipal solid waste incineration bottom ash (IBA), a major by-product of waste-to-energy plants, is typically landfilled or utilized as low-grade aggregate due to its low intrinsic reactivity and complex composition. This study systematically investigates the efficacy of chemical pretreatment in enhancing the cementitious behavior of IBA, specifically examining the effects of alkali type ($\text{Ca}(\text{OH})_2$, NaOH , and Na_2CO_3) and pretreatment duration on reactivity, microstructure, and mechanical performance. The results indicate that $\text{Ca}(\text{OH})_2$ activation provides the most significant enhancement; a one-day treatment yielded a 28-day strength activity index (H_{28}) of 76% and facilitated the formation of a compact microstructure rich in ettringite (Aft) and C-S-H gels. Conversely, NaOH and Na_2CO_3 treatments were less effective, leading to increased porosity and reduced strength attributed to charge imbalance and excessive carbonation, respectively. Prolonged alkaline treatment yielded diminishing returns, causing premature gel densification or excessive silicate depolymerization. Life-cycle assessment (LCA) revealed that Na_2CO_3 pretreatment entails the highest carbon footprint due to its high molar mass and energy-intensive production, whereas NaOH offers the highest CO_2 efficiency per unit of reactivity. Overall, $\text{Ca}(\text{OH})_2$ represents a balanced strategy, combining strong activation potential, chemical compatibility, and moderate carbon emissions, thereby supporting the sustainable valorization of IBA in low-carbon cementitious systems.

Keywords: incineration bottom ash (IBA); chemical pretreatment activation; life-cycle assessment; pore structure



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1. Introduction

With the accelerated pace of global urbanization and population growth, the generation of municipal solid waste has been steadily increasing. Traditional landfill disposal methods are becoming unsustainable due to the depletion of available land resources and the growing risk of secondary environmental pollution [1,2]. As a result, municipal

solid waste incineration (MSWI) has emerged as a preferred treatment technology in many regions because of its efficiency in reducing waste volume and recovering energy [3–5]. However, the by-products generated from incineration, particularly municipal solid waste incineration bottom ash (IBA), present new environmental and technical challenges. It is estimated that bottom ash constitutes 70–80% of the total solid residues from the incineration process [6,7]. IBA typically contains a mixture of metallic oxides, silicates, aluminates, and residual unburned materials. When improperly managed through landfilling or open storage, these residues can lead to heavy metal leaching and groundwater contamination [8,9]. Consequently, the resource utilization of IBA has become an urgent research focus for its use as a type of construction material.

However, the intrinsic reactivity of untreated IBA is relatively low because of its high content of inert glassy phases and incomplete vitrification during incineration [10,11]. Consequently, most engineered applications employ IBA as coarse or fine aggregate in concrete and pervious pavements after tailored pretreatments (e.g., washing, grading, or grinding) to mitigate deleterious components [12,13]. Concurrently, researchers are exploring alternative valorization routes, including activation for use as supplementary cementitious materials (SCM) [14–16], alkali activation/geopolymerization [17,18], and resource-recovery schemes, which aim to balance technical feasibility, environmental safety, and economic viability beyond simple aggregate substitution. Nevertheless, the high heterogeneity of IBA and the presence of metallic Al and mobile heavy metals can impair hydration, increase porosity, or raise leaching risks [19,20]. Particularly, metallic Al in IBA reacts with the alkaline environment of cement to generate H_2 gas, leading to the deterioration of concrete performance [21].

Researchers have used several complementary measures to reduce H_2 gas risk and control dissolution kinetics [22–24]: (i) mechanical removal or size-classification to reduce metallic Al exposure; and (ii) lower alkali concentration, buffered solutions, or staged/short-duration soaking to limit rapid H_2 release. Among the pretreatments of IBA, chemical pretreatment of metallic Al has been widely used, serving two principal purposes: (i) the safe removal or chemical conversion of metallic Al, which reacts vigorously in alkaline media to produce hydrogen gas and internal porosity, and (ii) the controlled dissolution/activation of Al^{3+} and Si^{4+} bound in the glassy matrix, so that these species can participate in subsequent pozzolanic or alkali activation reactions. Conventional strong-alkali treatments (e.g., concentrated NaOH) have been widely used to pretreat IBA or produce aerated concrete [25–27].

However, conventional NaOH-based activation systems exhibit excessively high alkalinity, and the elevated hydroxyl ion concentration accelerates the dissolution of reactive silica within aggregates, increasing the risk of alkali–silica reaction (ASR) and consequent expansion or cracking in hardened material [28–30]. At the same time, metallic aluminum in incineration bottom ash (IBA) typically exists in the form of thin films or is encapsulated by oxide layers such as Al_2O_3 or $Al(OH)_3$. This passivation layer significantly reduces the reaction rate between metallic aluminum and alkaline solutions [31]. Only with prolonged alkaline treatment does the passivation layer gradually dissolve, allowing metallic Al to react with OH^- to form $Al(OH)_4^-$ and release hydrogen gas. Therefore, insufficient treatment time may result in some metallic Al continuing to react during cement hydration, leading to expansion and porosity issues [32].

Therefore, in this study, alternative or moderated alkaline activators, such as $Ca(OH)_2$ and Na_2CO_3 , that can possibly achieve sufficient activation of IBA while mitigating adverse effects on cement chemistry and long-term durability were explored. Prolonged pretreatment was also employed, which could enable gradual leaching and stabilization of metallic Al, reducing H_2 evolution during concrete specimen preparation. Furthermore,

the mechanical performance of concrete containing IBA with different types of alkali pretreatment and pretreatment durations was investigated, and the mechanism behind the strength variation was interpreted by the later microstructural and pore structural analysis. Finally, for chemical-treated IBA, assessing its carbon footprint requires a fair comparison with competing SCMs such as fly ash or slag. Traditional life-cycle carbon accounting may overestimate or underestimate the impact of IBA if differences in their reactivity are ignored. Therefore, carbon footprint evaluations should normalize emissions relative to the material's actual pozzolanic or hydraulic activity, ensuring that the performance-per-unit- CO_2 metric reflects the true environmental efficiency of the SCM, that is, chemical-pretreated IBA, fly ash, and slag.

Although previous studies have explored alkali activation of IBA for use as an SCM, most have focused on single activators or limited treatment conditions, lacking a systematic comparison of multiple alkaline types and durations coupled with integrated environmental assessment. The present work addresses this gap by introducing a comprehensive optimization framework that links activation efficiency, mechanical performance, and carbon footprint. Specifically, the novelty of this study lies in (1) the simultaneous evaluation of three distinct alkaline activators ($\text{Ca}(\text{OH})_2$, NaOH , and Na_2CO_3) across a wide range of pretreatment durations (1–10 days) to identify non-linear treatment effects; (2) the integration of advanced microstructural characterization techniques, notably X-ray computed tomography (X-CT), with traditional pore analysis to provide three-dimensional insight into pore network evolution; and (3) the development of a reactivity-normalized CO_2 intensity metric that quantitatively balances mechanical enhancement with environmental impact, offering a holistic criterion for sustainable SCM selection. By bridging performance optimization with carbon-efficiency assessment, this research provides a systematic pathway for the low-carbon valorization of IBA, advancing both technical and environmental dimensions of waste-derived cementitious materials.

2. Materials, IBA Pretreatment, Mortar Preparation, and Method

2.1. Materials

The cement utilized in this experiment was 42.5-grade ordinary Portland cement (OPC) provided by Hangzhou Dingsheng Building Materials Co., Ltd. (Hangzhou, China). The IBA utilized in this experiment was sourced from the municipal solid waste incineration facility in Shaoxing City, with a moisture content of 0.069%. The chemical compositions of OPC and IBA were analyzed using XRF, as presented in Table 1. The particle size properties were assessed using a laser particle size analyzer, and the specific surface area and pore size were measured using an analyzer from Micromeritics Instrument Corp. (Shanghai, China), as shown in Figure 1.

Table 1. Chemical compositions of OPC and MSWIBA.

Composition (%)	CaO	SiO ₂	Al ₂ O ₃	SO ₃	Fe ₂ O ₃	K ₂ O	TiO ₂	MgO	Cl	P ₂ O ₅
OPC	41.5	22.10	8.63	3.92	3.73	0.92	0.44	0.18	0.11	0.13
IBA	22.63	25.73	8.95	5.65	27.23	2.04	1.48	3.92	0.25	2.11

The fine aggregate utilized in this experiment was ISO standard sand manufactured by Xiamen AISO Standard Sand Co., Ltd. (Xiamen, Chian), in compliance with GB/T17671-2021 [33]. The water specimens utilized in this experiment were sourced from the laboratory's municipal water supply system. The alkaline-stimulating reagents employed in this study were all Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) reagents, procured from China National Pharmaceutical Group Chemical Reagent Co., Ltd. (Shanghai, China)

These included sodium hydroxide (NaOH; Purity: $\geq 95.0\%$), sodium carbonate (Na_2CO_3 ; Purity: $\geq 99.8\%$), and calcium hydroxide ($\text{Ca}(\text{OH})_2$; Purity: $\geq 96\%$).

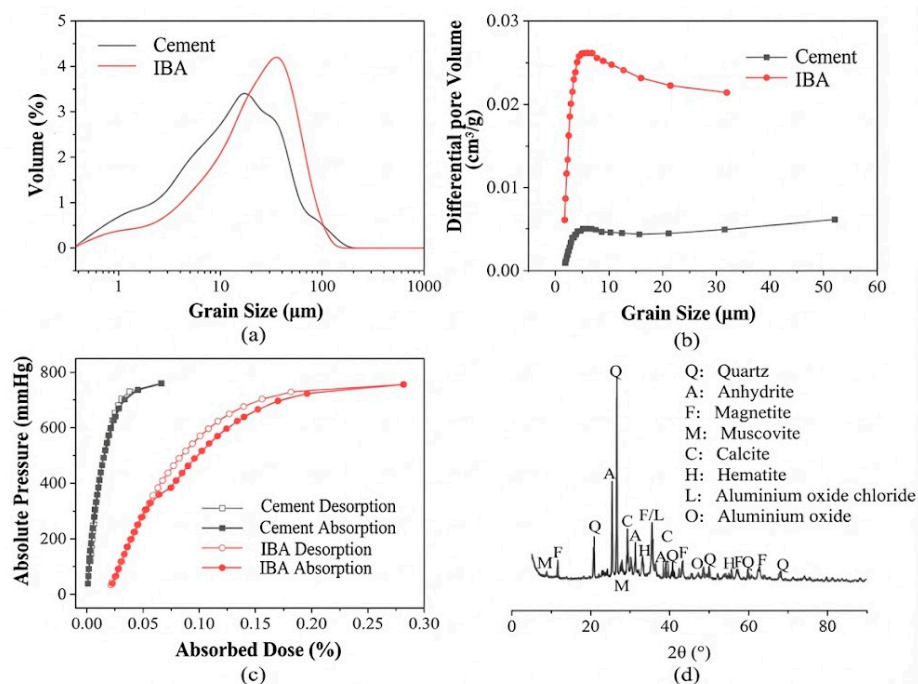


Figure 1. (a) Particle size distribution of IBA and cement; (b) pore size distribution of IBA and cement; (c) specific surface area of IBA and cement; and (d) XRD pattern of IBA.

2.2. IBA Pretreatment and Mix Formulation

Optimization of the chemical activation pretreatment method involves enhancing the reactivity in IBA through chemical processes. Research indicates that an alkaline environment can dissolve the amorphous phase in IBA, releasing soluble silica and alumina, which subsequently contribute to the development of hydration products such as C-S-H gel and calcium aluminate during the hydration process.

The impact of soaking duration on the activating effect of IBA was examined by immersing it in various alkaline solutions. In the experiment, IBA was immersed in 0.2 mol/L solutions of $\text{Ca}(\text{OH})_2$, NaOH, and Na_2CO_3 , with a solid-liquid ratio of 1:5, for durations of 1, 3, 5, 7, and 10 days. The use of $\text{Ca}(\text{OH})_2$ was intended to supplement the lack of an active calcium source in IBA by directly providing Ca^{2+} to promote the nucleation of the C-S-H gel, thereby mimicking the conventional pozzolanic reaction mechanism. In contrast, NaOH represents a typical strong alkali activation approach aimed at breaking the vitreous network (Si-O-Si bonds) through high pH, while simultaneously introducing sodium ions that may potentially interfere with the gel structure. Na_2CO_3 , as a low-cost and commonly available industrial alkali, was employed to examine whether a “weak alkali + carbonate” system could fill pores through the formation of carbonate precipitates, despite its relatively lower pH. Regarding the time span (1–10 days), our objective was to capture the transition from surface cleaning to deep dissolution [5]. Experimental results confirmed that prolonged immersion (>3 days) did not lead to linear improvement; instead, it resulted in compromised performance due to over-etching or the formation of a passive layer, thereby validating the economic and technical effectiveness of short-term treatment. In order to systematically evaluate the effect of alkaline excitation parameters on IBA performance, this study adopted a 3×5 full factorial design, as shown in Table 2. Throughout the soaking procedure, it was stirred every 12 h and sealed to avert CO_2 contamination to impregnate alkali on IBA. Subsequent to soaking, the IBA was subjected

to filtration and was washed twice with laboratory water to completely eliminate the leftover alkaline solution. The purified IBA was subsequently placed in an oven at 60 °C and dried for 24 h. The procedures of IBA chemical pretreatment are shown in Figure 2. The specimen nomenclature follows a systematic rule to indicate both the type of alkaline activator and the activation duration, and “M30” represents 30% of OPC is replaced by IBA. Specifically, the prefix represents the activator used: “CH” for 0.2 mol/L $\text{Ca}(\text{OH})_2$, “NH” for 0.2 mol/L NaOH, and “NC” for 0.2 mol/L Na_2CO_3 . The middle number (1 d, 3 d, 5 d, 7 d, or 10 d) denotes the activation period in days. For instance, “M30-CH-5d” indicates mortar contains 30% of IBA soaked in 0.2 mol/L $\text{Ca}(\text{OH})_2$ solution for 5 days at a temperature of 20 °C, and the liquid to solid ratio of alkali is 5. Table 3 shows the nomenclature of different groups. The group C100 was designated as the control group comprising pure cement. A portion of cement was substituted with 30% MSWIBA, forming the group M30. The mix proportion of mortar in this study is as follows: 315 g of ordinary Portland cement (OPC), 135 g of IBA, and 1350 g of sand, with a w/c of 0.5.

Table 2. Experimental full factorial design matrix.

Alkali Activator (Factor A) \ Treatment Duration (Factor B)	1 Day (B1)	3 Days (B2)	5 Days (B3)	7 Days (B4)	10 Days (B5)
$\text{Ca}(\text{OH})_2$	M30-CH-1d	M30-CH-3d	M30-CH-5d	M30-CH-7d	M30-CH-10d
NaOH	M30-NH-1d	M30-NH-3d	M30-NH-5d	M30-NH-7d	M30-NH-10d
Na_2CO_3	M30-NC-1d	M30-NC-3d	M30-NC-5d	M30-NC-7d	M30-NC-10d

Table 3. The nomenclature of different groups.

Group	Alkali Type	Concentration	Temperature	Soaking Time	L/S Ratio
M30-CH-1d	$\text{Ca}(\text{OH})_2$	0.2 mol/L	20 °C	1 d	5
M30-CH-3d	$\text{Ca}(\text{OH})_2$	0.2 mol/L	20 °C	3 d	5
M30-CH-5d	$\text{Ca}(\text{OH})_2$	0.2 mol/L	20 °C	5 d	5
M30-CH-7d	$\text{Ca}(\text{OH})_2$	0.2 mol/L	20 °C	7 d	5
M30-CH-10d	$\text{Ca}(\text{OH})_2$	0.2 mol/L	20 °C	10 d	5
M30-NH-1d	NaOH	0.2 mol/L	20 °C	1 d	5
M30-NH-3d	NaOH	0.2 mol/L	20 °C	3 d	5
M30-NH-5d	NaOH	0.2 mol/L	20 °C	5 d	5
M30-NH-7d	NaOH	0.2 mol/L	20 °C	7 d	5
M30-NH-10d	NaOH	0.2 mol/L	20 °C	10 d	5
M30-NC-1d	Na_2CO_3	0.2 mol/L	20 °C	1 d	5
M30-NC-3d	Na_2CO_3	0.2 mol/L	20 °C	3 d	5
M30-NC-5d	Na_2CO_3	0.2 mol/L	20 °C	5 d	5
M30-NC-7d	Na_2CO_3	0.2 mol/L	20 °C	7 d	5
M30-NC-10d	Na_2CO_3	0.2 mol/L	20 °C	10 d	5

The selection of 0.2 mol/L as the alkali concentration was based on a balance between activation efficacy and volumetric stability. Higher alkali concentrations (e.g., >0.5 mol/L) may induce vigorous reaction of metallic aluminum present in IBA, leading to excessive hydrogen gas evolution and detrimental porosity [34,35]. The 0.2 mol/L threshold was thus chosen as a safe yet effective concentration to promote dissolution of amorphous phases without compromising microstructural integrity.

The 30% cement replacement level was adopted, as it represents a critical tipping point for SCMs, beyond which the dilution effect often outweighs the pozzolanic contribution, and the sensitivity to pretreatment efficacy becomes more pronounced [36]. This replacement ratio allows a clear evaluation of IBA's reactivity enhancement while maintaining reasonable workability and mechanical performance.

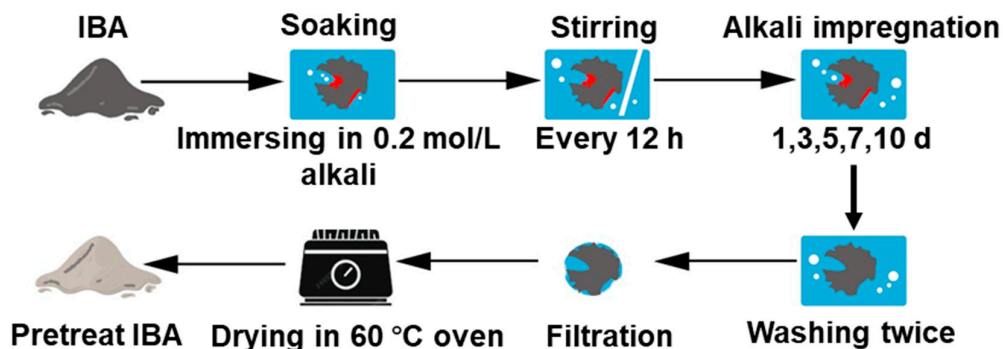


Figure 2. Procedures of IBA chemical pretreatment.

2.3. Mortar Preparation

The preparation technique for the mortar specimens adhered rigorously to the experimental design. The necessary cementitious materials (OPC and IBA) were measured according to the mix ratio and meticulously combined using a stirring rod. Pre-weighed $\text{Ca}(\text{OH})_2$, NaOH, and Na_2CO_3 were solubilized in water and reserved.

During preparation, water or the formulated solution was introduced into the mixing vessel. The OPC and IBA were subsequently introduced, and the mortar mixer was promptly activated for 30 s. At the commencement of the subsequent 30 s interval, sand was uniformly introduced into the pot, and agitation persisted for 30 s. Stirring ceased for 90 s, during which mortar was removed from the mixing blades and the pot walls and collected into the pot. Stirring was prolonged for an extra 60 s to guarantee homogeneous mixing. Subsequent to mixing, the mortar was deposited into two oil-coated molds measuring $40 \times 40 \times 160 \text{ mm}^3$ and $40 \times 40 \times 40 \text{ mm}^3$, respectively. Initially, the mold was filled halfway with mortar and crushed on a vibrating table for 30 s. The mold was subsequently filled with mortar and crushed for a further 30 s. Following compaction, the surface was leveled with a spatula and subsequently covered with a film for preliminary curing. Following a 24 h period, the specimens were extracted from the molds and transferred to a conventional curing room, where they were subjected to a CO_2 concentration of 0.04%, a relative humidity of 60%, and a temperature of 20 °C for curing. Upon reaching the designated curing age, the specimens were extracted, and performance evaluations were conducted.

2.4. Methodology

2.4.1. Experimental Method of Isothermal Calorimetry

Isothermal calorimetry was employed to assess the hydration behaviors of IBA by monitoring its heat release. The experiments were performed in a TAM Air isothermal calorimeter (TA Instruments, New Castle, DE, USA), maintaining an ambient temperature of 60 °C with a control precision of ± 0.1 °C. A curing temperature of 60 °C was employed in isothermal calorimetry to accelerate the otherwise slow reaction kinetics of IBA, enabling observable heat evolution within a practical experimental timeframe. This approach aligns with ASTM C1074 [37] principles and previous studies on low-reactivity SCMs, where elevated temperatures are used to discern hydration characteristics under accelerated conditions [38]. During specimen preparation, IBA, cement, and deionized water were

combined in a ratio of 3:7:4 and thereafter transferred to a test cell without delay. The examination went for 72 h. The system autonomously documented parameters, including the hydration heat release rate and total heat release. Concurrent specimens were employed for each experimental set to guarantee dependable results.

2.4.2. Reactivity Test

Although IBA's chemical composition is similar to that of cement, its reactivity is significantly inferior to that of ordinary Portland cement. To measure the activation effects of different pretreatment methods, the strength-reactivity index H_{28} is used for quantitative evaluation in accordance with the Chinese national standard GB/T 1596-2017 [39]. The reactivity index was calculated using Equation (1).

$$H_{28} = R/R_0 \times 100\% \quad (1)$$

H_{28} represents the reactivity index, rounded to the nearest 1%. R denotes the 28 d compressive strength (MPa) of cement mortar in which 30% of the cement is replaced by IBA, while R_0 denotes the 28 d compressive strength (MPa) of the control cement mortar.

2.4.3. Mechanical Performance

Compressive and flexural strength assessments were conducted on specimens at various curing durations (3, 14, and 28 days). A YDW-300C servo testing machine (Jinhua Julong Computer Testing Machine Co., Ltd., Jinhua, China) was employed for compressive and flexural loads at rates of 10 kN/min and 20 kN/min, respectively. The testing technique rigorously complied with the GB/T 17671-2021 standard [33]. Three specimens were employed for each test, with the average and standard deviation calculated as the final results.

2.4.4. XRD, TG/DTG, FTIR, and SEM

The specimen preparation technique rigorously conformed to microanalysis standards: The specimen, aged as required, was crushed, and interior fragments roughly 5 mm in size, with a surface as flat as feasible, were picked from about 10 mm beneath the surface. Hydration was concluded by immersion in anhydrous ethanol for 3 days, followed by drying in a 60 °C oven until a constant weight was achieved (mass change $\leq 0.05\%$) prior to XRD, TG/DTG, FTIR, and SEM.

X-ray diffraction (XRD) is a crucial method for analyzing the composition and development of hydration products in cementitious materials. Examining the mineral composition and content of specimens at various ages (3 and 28 days) establishes a foundation for investigating the evolutionary mechanisms of the material's macroscopic and microscopic characteristics. Internal specimens (about 10 mm from the surface) were retrieved from mortar specimens cured to the appropriate age during the experiment. The specimens were submerged in anhydrous ethanol for three days to halt the hydration reaction. The specimens were subsequently dried at 60 °C until a consistent weight was achieved, crushed, and sieved through a 0.075 mm square mesh to prepare the test powder.

The experiments were performed utilizing a Bruker D8 Advance diffractometer (Bruker AXS GmbH, Karlsruhe, Germany) with a Cu-K α radiation source (40 kV, 40 mA), covering a scanning range of $2\theta = 5^\circ$ to 90° , in increments of 0.02° . Quantitative phase analysis of the XRD patterns was performed using the Rietveld refinement method to determine the weight fractions of crystalline phases. The refinement was conducted using the Jade 6.5 software with the fundamental parameters approach. To improve accuracy, 10 wt.% of α -Al₂O₃ (corundum, NIST SRM 676a) was added as an internal standard to each sample prior to analysis. The crystal structure models for the identified phases (e.g., ettringite, portlandite, calcite, and gehlenite) were obtained from the Inorganic Crystal

Structure Database (ICSD) [40]. The refinement process included adjustments for scale factors, background (Chebyshev polynomial), unit cell parameters, and microstructure (e.g., crystallite size and microstrain) for major phases until a satisfactory fit was achieved (goodness-of-fit indicator, $R_{wp} < 10\%$) [41,42].

The phase percentages reported and discussed in this research are derived from the refined scale factors. The uncertainty associated with these quantified values is estimated to be within $\pm 1\text{--}2$ wt.% for major phases (e.g., Aft and portlandite). This uncertainty primarily arises from instrumental factors (counting statistics and background subtraction), the complexity of the multi-phase system containing amorphous content, and the limitations of the crystal structure models for minor or poorly crystalline phases. The reported difference in Aft content (e.g., 2.1% between M30-CH-1d and M30-CH-10d) is therefore considered significant, as it exceeds the typical margin of error associated with the method.

Thermogravimetric analysis (TG/DTG) is a technique for quantitatively assessing the composition of hydration products by examining variations in the decomposition temperature of a material. The results can augment XRD data. The experiment was performed on a Mettler Toledo STARe-System TGA2 (Mettler Toledo, Schwerzenbach, Switzerland) in a nitrogen environment. The parameters were established with a heating rate of $10\text{ }^{\circ}\text{C}/\text{min}$ and a temperature range from $30\text{ }^{\circ}\text{C}$ to $1000\text{ }^{\circ}\text{C}$. The temperature-dependent mass curve of the test powder was recorded, quantitatively characterizing the decomposition process and the amount of various mineral components, thereby offering significant evidence for a comprehensive investigation into material composition and qualities.

Fourier transform infrared spectroscopy (FTIR) enables qualitative and quantitative analyses of a specimen's chemical structure and composition by identifying the unique absorption frequencies of functional groups present in a molecule. The examination was conducted utilizing a NICOLET iS50 FT-IR spectrometer (Thermo Scientific, Waltham, MA, USA), featuring a spectrum range of $4000\text{--}400\text{ cm}^{-1}$ and a resolution of 1 cm^{-1} . The analysis of the infrared spectra of the specimen identifies the types and quantities of functional groups present in the material. The dried specimen was subsequently deduced (purged with argon) to eliminate surface impurities and enhance conductivity.

2.4.5. MIP and X-CT Analysis Method

Mercury intrusion porosimetry (MIP) is a proficient technique for examining the pore architecture of cementitious materials, elucidating the inherent correlation between pore structure and macroscopic properties. The test utilized a fully automated mercury intrusion porosimeter, the Autopore IV 9500 (Micromeritics, Norcross, GA, USA). The precise steps involved are as follows: Following 3 and 28 days of curing, specimens were extracted from the specimen's interior (about 10 mm from the surface) and submerged in anhydrous ethanol for 3 days to halt the hydration reaction. The specimens were subsequently dried at $60\text{ }^{\circ}\text{C}$ until a consistent weight was achieved and then comminuted into particles measuring 2–3 mm for MIP testing. Mercury intrusion porosimetry yields total porosity and pore size distribution characteristics of specimens at various ages, offering a microscopic foundation for comprehending the evolution of mortar qualities.

While mercury intrusion porosimetry offers quantitative insights into overall porosity and pore size distribution, it is challenging to comprehensively characterize the intricate three-dimensional pore architecture. X-ray computed tomography (X-CT) addresses this constraint by facilitating nondestructive three-dimensional visualization and quantitative study of interior pore structure, offering a precise method for characterizing the pore structure of cementitious materials. This investigation utilized a Nikon XTH320 X-ray computed tomography scanner (Nikon Corp., Tokyo, Japan) to scan and image the specimens. The scanning settings were established at 159 kV and 120 μA . Two thousand projections were

obtained during a 360° rotation of each specimen, with an exposure duration of 0.5 s per projection. Following scanning, 2D tomographic images were rebuilt via CTPro 2.1 software, and pore analysis was conducted with VGStudio software through grayscale thresholding. The experiment concentrated on the interior architecture of specimens aged 28 days. The pore structure was ascertained by selecting a $3 \times 3 \times 3 \text{ mm}^3$ region of interest, employing adaptive Gaussian filtering for noise reduction and a grayscale threshold technique. The “Defect Analysis” algorithm in VGStudio Max 3.0 software was employed to acquire the characteristics, including pore radius and volume. This demonstrated the impact of several IBA activation techniques on the pore structure of the material. The integration of MIP and X-CT analysis results thoroughly examines the relationship between pore structure characteristics and material properties, guaranteeing an accurate evaluation of the pore structure in IBA-containing cementitious materials.

2.4.6. Carbon Footprint Assessment

The purpose of conducting the carbon footprint assessment of pretreated IBA, fly ash, and slag is to quantitatively evaluate their potential environmental impacts when used as SCMs. By analyzing the life-cycle CO_2 emissions associated with their production and treatment, this assessment aims to identify the relative CO_2 emissions of each SCM. Therefore, it also provides a scientific basis for optimizing SCM selection (~pretreated IBA, fly ash, and slag) and developing low-carbon concrete mixtures that balance performance and sustainability.

At the same time, by correlating the CO_2 emissions achieved through chemical pretreatment with the improvement in reactivity of IBA, this assessment helps identify the selection of the optimal alkali type that maximizes both the sustainability and functionality of IBA. Such an integrated approach provides scientific support for the large-scale utilization of IBA as a low-carbon SCM in cement and concrete production.

The functional unit is to produce 1 kg of IBA, fly ash, and slag. The assessment follows a cradle-to-gate approach, with the system boundary encompassing raw material acquisition, transportation to the pretreatment facility, and the chemical pretreatment process itself (as delineated by the dotted line in Figure 2). Specifically included are: the production of the alkaline reagents (NaOH , $\text{Ca}(\text{OH})_2$, and Na_2CO_3) and their transportation (assuming a default distance of 100 km by truck), the electricity for stirring during immersion, and the thermal energy for drying the treated IBA. Key exclusions are: capital goods (e.g., reactor infrastructure), transportation of raw IBA from the incineration plant (as it is considered a waste with zero burden at the gate), laboratory water use, and the final mortar mixing and curing processes. This boundary focuses on the incremental environmental burden imparted by the chemical activation step, enabling a direct comparison of the activation strategies. For IBA, the environmental impacts are ascribed to its pretreatment, and the system boundary of IBA pretreatment (as circled in a dotted line) in this study is shown in Figure 3. To pretreat IBA, as the liquid to IBA mass ratio is 5, solution density is assumed to be $1000 \text{ kg}\cdot\text{m}^{-3}$ ($1 \text{ kg} \approx 1 \text{ L}$), alkali concentration = $0.200 \text{ mol}\cdot\text{L}^{-1}$, and the amount of alkali used is Na_2CO_3 : $\approx 106.0 \text{ g}$; $\text{Ca}(\text{OH})_2$: $\approx 74.1 \text{ g}$; and NaOH : $\approx 40.0 \text{ g}$. The life-cycle inventories of the corresponding alkali are from the database Ecoinvent. For the pretreatment of fly ash and slag, their life-cycle inventories are derived from the literature [43].

In this study, the CML2000 method was chosen to evaluate the global warming potential related to the production of SCMs, as expressed by the emitted CO_2 -equivalent. All the calculations are performed with SimaPro 9.5.0 to evaluate the carbon footprint for comparison. By simultaneously assessing CO_2 emissions (C_0) associated with pretreatment and the resulting improvement in reactivity (H_{28}), this approach identifies the most efficient

alkali that achieves the lowest carbon impact while maximizing IBA reactivity, which is expressed as CO₂ intensity (C_i) (Equation (2)).

$$C_i = C_0 / H_{28} \times 100\% \quad (2)$$

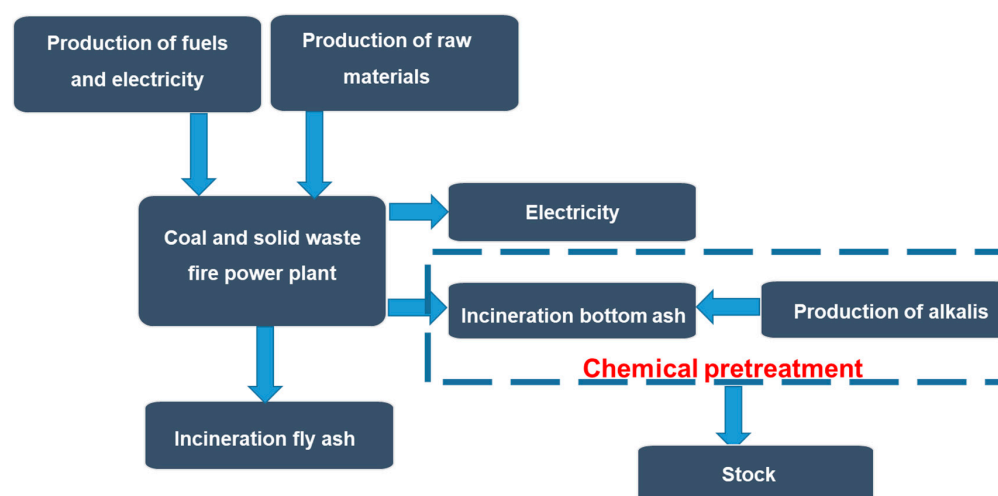


Figure 3. The system boundary of the IBA pretreatment.

3. Result and Discussion

3.1. Isothermal Calorimetry

Figure 4a illustrates the hydration rate curves of pure cement (C100 group) and the system with 30% cement replaced by chemically pretreated IBA. The variations in hydration behavior can be attributed to the differences in chemical composition shown in Table 4. The hydration behaviors can be divided into the following stages:

Table 4. Chemical compositions of IBA groups after chemical pretreatment.

Composition (%)	CaO	SiO ₂	Al ₂ O ₃	SO ₃	Fe ₂ O ₃	K ₂ O	TiO ₂	MgO	Cl	P ₂ O ₅
CH-1d	27.94	22.31	9.33	5.52	27.28	1.68	1.25	2.87	0.33	1.50
CH-3d	27.45	21.53	9.40	5.37	27.64	2.00	1.33	3.34	0.27	1.66
CH-5d	27.06	21.99	9.07	5.74	27.75	1.96	1.69	2.92	0.29	1.51
CH-7d	26.83	22.63	8.92	5.70	27.40	2.32	1.28	3.07	0.28	1.57
CH-10d	26.89	22.94	9.10	5.76	27.21	1.97	1.23	3.04	0.28	1.58
NH-1d	24.98	23.02	9.95	3.36	30.01	1.92	1.50	3.11	0.32	1.73
NH-3d	24.64	23.11	9.42	3.33	30.49	2.12	1.43	3.87	0.29	1.94
NH-5d	24.56	23.70	8.87	3.35	30.18	2.06	1.55	3.52	0.32	1.9
NH-7d	24.22	24.31	8.66	3.22	30.80	1.83	1.42	3.46	0.31	1.77
NH-10d	24.18	24.53	8.64	3.30	30.33	1.81	1.40	3.61	0.31	1.90
NC-1d	23.10	25.17	9.28	4.78	28.02	2.20	1.52	3.68	0.24	2.00
NC-3d	23.03	25.24	9.33	4.07	28.40	2.19	1.54	3.83	0.27	2.11
NC-5d	22.69	25.43	9.01	4.14	28.79	2.18	1.55	3.83	0.28	2.09
NC-7d	22.51	26.03	8.98	4.12	28.47	2.23	1.49	3.78	0.26	2.11
NC-10d	22.08	26.01	9.02	4.95	28.72	1.87	1.40	3.76	0.25	1.95

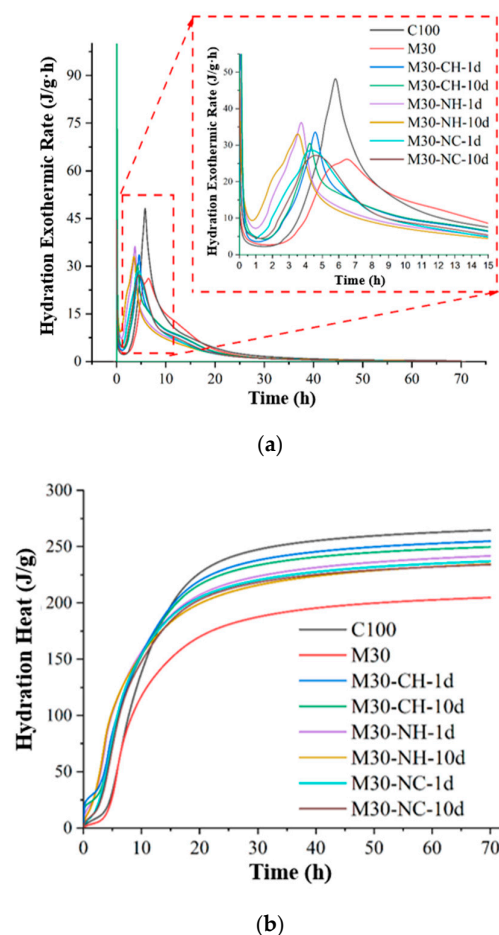


Figure 4. (a) Hydration rate and (b) accumulative hydration heat of selected groups with different types of pretreated IBA.

(1) The early reaction period (~0–40 min) revealed that all groups exhibited significant exothermic peaks, with the C100 group displaying the largest peak and the M30 group displaying the lowest. The pronounced exothermic peak of the C100 group resulted from its elevated CaO content, which facilitated the fast reaction of C_3A with gypsum to produce calcium aluminate [43]. In the M30 group, the reduced calcium concentration (Table 4) in IBA inhibited the hydrolysis of C_3S , while the elevated Fe^{3+} levels depleted OH^- in the pore solution via oxidation reactions, hence further postponing the hydration process.

(2) Subsequent to the induction period (~40–90 min), the reaction rate diminished markedly. The induction duration for the C100 group was approximately 60 min, but the M30 group experienced an extension to 90 min. The prolongation of the induction period in the M30 group is attributed to the high concentrations of SO_3 and Al_2O_3 in IBA. The dissolution of these species releases SO_4^{2-} and Al^{3+} , which suppress the hydration of C_3S and extend the period of slow reaction [44]. In the groups subjected to pretreated chemical activation, the soluble compounds on the IBA surface were largely dissolved during the pretreatment phase, which diminished the inhibitory effect on C_3S dissolution, leading to a decreased induction duration.

(3) The heat release rate throughout the acceleration phase (~1.5–20 h) was regulated by the nucleation and development of the C-S-H gel, mostly influenced by C_3S . The elevated levels of Fe_2O_3 and Al in the M30 group led to the continuous consumption of OH^- by Fe^{3+} and Al to form precipitates, which delayed the rise in the pH of the pore solution, thereby markedly limiting the hydrolysis of C_3S and resulting in the lowest rate of heat release. Conversely, the NH-1d group stimulated amorphous SiO_2 and Al_2O_3 (Table 4)

to produce N-A-S-H, significantly enhancing the exothermic rate. However, prolonged alkali treatment resulted in excessive dissolution of the active components in IBA, leading to inferior mechanical properties (Section 3.2) of the resultant sodium silicate gel, which caused a rightward shift of the exothermic peak and a reduction in its peak value. The elevated Ca^{2+} levels in the CH-1d group directly facilitated the nucleation of the C-S-H gel, thereby enhancing the exothermic rate. Conversely, the NC-1d group exhibited the least improvement in exothermic rate due to the restricted activation efficacy of IBA in the mildly alkaline environment of Na_2CO_3 .

(4) Following the deceleration phase (~20–48 h) and the steady reaction phase (~48–72 h), the reaction rate was governed by ion diffusion [45]. The difference between the groups progressively diminished; however, the tendency remained considerable. The M30-CH-1d group exhibited sustained reactivity owing to the persistent release of Ca^{2+} (Table 4). On the contrary, the M30-NH-1d group experienced a notable decrease in heat release during the later phase due to the swift depletion of active components in the initial phase, resulting in a marked reduction in the subsequent hydration heat release rate.

Figure 4b illustrates the cumulative heat of hydration for each group for a duration of 72 h. The C100 group revealed the greatest total cumulative heat of hydration (~265 J/g), correlating with its elevated concentration of active components. The M30-CH-1d group had a cumulative heat of 255 J/g, which is nearest to the C100 group, implying the most substantial enhancement in IBA reactivity. The M30-NH-10d group exhibited a cumulative heat of merely 234 J/g, corroborating the deleterious impact of prolonged strong alkaline treatment on IBA. The M30 group had the lowest cumulative heat of hydration (~205 J/g), attributable to its reduced CaO and elevated Fe_2O_3 concentration, as shown in Table 4.

3.2. Reactivity and Mechanical Performance

Figure 5a–d show that the compressive strength and corresponding H_{28} values of samples at different curing ages were measured. The results indicate that both the type of alkaline solution and immersion duration have a significant influence on the compressive strength of mortars incorporating 30% IBA as a cement replacement. Overall, samples treated with $\text{Ca}(\text{OH})_2$ solution (M30-CH) exhibited the highest strength, followed by those treated with NaOH (M30-NH), while Na_2CO_3 -treated samples (M30-NC) showed the weakest improvement. Generally, compressive strength exhibited an initial increase followed by a decline with prolonged immersion duration. Figure 5e–g present the flexural strength evolution, which follows a trend consistent with the compressive strength results.

For the $\text{Ca}(\text{OH})_2$ -treated group, the figures show that M30-CH-1d achieved a 28-day compressive strength of 36.63 MPa and a flexural strength of 8.25 MPa, with an H_{28} reactivity value of 76%. This enhancement was attributed to the short-term $\text{Ca}(\text{OH})_2$ immersion, which supplements Ca^{2+} to directly promote C-S-H gel nucleation [46], while forming a surface CaCO_3 coating that mitigates interference from aluminum oxidation during hydration, thereby improving cementitious efficiency. However, after 10 days of immersion, the M30-CH-10d sample exhibited decreased compressive and flexural strengths of 33.92 MPa and 7.42 MPa, respectively. This decline likely results from excessive leaching or passivation of active components in IBA, reducing the sustained release of calcium ions. Therefore, one-day immersion in $\text{Ca}(\text{OH})_2$ solution is identified as the optimal pretreatment condition.

For NaOH-treated samples, the strength enhancement was moderate. The M30-NH-1d sample reached a compressive strength of 34.23 MPa and a flexural strength of 8.06 MPa. This can be attributed to the strong alkalinity, which activates amorphous aluminosilicate phases in IBA, promoting partial gel formation. However, the M30-NH-10d sample showed reduced strengths (33.41 MPa and 7.59 MPa), likely due to over-dissolution of reactive

species under prolonged alkaline exposure, leading to the formation of mechanically weaker N-A-S-H gels [47]. These results highlight the importance of controlling immersion duration to avoid activity loss during NaOH pretreatment.

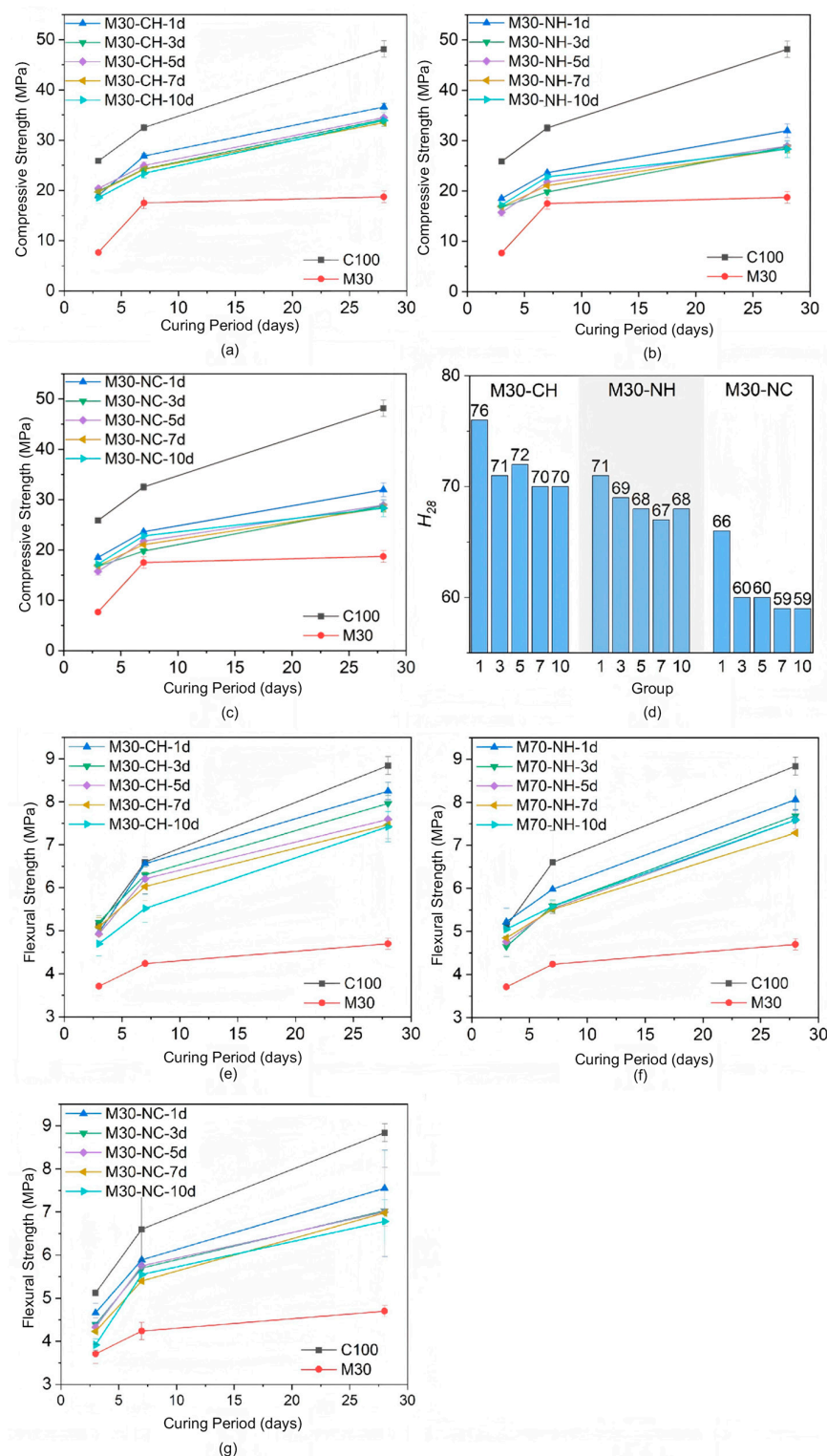


Figure 5. Compressive strength of specimens up to 28 days: (a) M30-CH group; (b) M30-NH group; (c) M30-NC group; and (d) reactivity (H₂₈) of specimens in each group and flexural strength of specimens: (e) M30-CH group; (f) M30-NH group; and (g) M30-NC group.

In contrast, Na₂CO₃ treatment resulted in the weakest strength improvement. The M30-NC-1d sample achieved a compressive strength of only 31.96 MPa and a flexural

strength of 7.55 MPa, slightly higher than the untreated M30 sample but far below that of the $\text{Ca}(\text{OH})_2$ group. This is because the weak alkalinity of Na_2CO_3 provides limited activation, and the rapid precipitation of CaCO_3 consumes available Ca^{2+} , thereby inhibiting the nucleation and growth of the primary binding phase, C-S-H gel [48], decreasing available calcium and hindering C-S-H gel formation. Even after 10 days of immersion, the compressive strength remained below 30 MPa, indicating that Na_2CO_3 treatment is ineffective in enhancing IBA reactivity.

Figure 5e–g illustrate the development of flexural strength for each group: (a) M30-CH, (b) M30-NH, (c) M30-NC, and (d) comparison of 1-day immersion across different activators. The effect of immersion duration on compressive strength exhibits a clear non-linear pattern. For all alkaline solutions, the optimal performance was achieved after one day of immersion, while longer durations (3–10 days) led to slight strength reduction. This suggests that short-term immersion effectively removes soluble impurities and activates latent cementitious phases in IBA, whereas prolonged immersion causes excessive leaching or passivation, weakening its synergy with cement hydration.

In summary, compared to the controls, the chemically pretreated groups showed improved mechanical properties. The C100 (pure cement) group yielded the highest strength, while M30 (30% untreated IBA) exhibited the lowest due to dilution and interference. As shown in Table 5, among the pretreated groups, M30-CH-1d performed best, achieving 36.63 MPa at 28 days and an H_{28} of 76%, significantly surpassing M30 ($p < 0.01$) and approaching C100 performance. $\text{Ca}(\text{OH})_2$ pretreatment was more effective than NaOH or Na_2CO_3 , with short-term (1 day) treatment optimally supplementing Ca^{2+} and promoting C-S-H/AfT formation. NaOH showed moderate improvement, while Na_2CO_3 provided limited benefit due to weak activation and carbonation. In summary, chemical pretreatment enhances IBA reactivity, with 1-day $\text{Ca}(\text{OH})_2$ treatment offering the best balance between performance and resource utilization.

Table 5. Statistical processing of 28-day compressive strength data for selected groups.

Group	28d Compressive Strength (MPa)	Standard Deviation (SD)	Coefficient of Variation (COV, %)	Improvement Rate Comparing with M30	<i>p</i> -Value (ANOVA)
C100	48.20	1.52	3.15	+69.1%	-
M30	28.50	1.18	4.14	Ref.	-
M30-CH-1d	36.63	0.95	2.59	+28.5%	<0.01
M30-CH-10d	33.92	1.45	4.27	+19.0%	<0.05
M30-NH-1d	34.23	1.82	5.32	+20.1%	<0.05
M30-NH-10d	33.41	2.10	6.29	+17.2%	<0.05
M30-NC-1d	31.96	3.48	10.89	+12.1%	>0.05

3.3. XRD

Figure 6 illustrates the XRD diffraction patterns of selected groups after being cured for 3 and 28 days, whereas Figure 7 presents the ratios of each mineral phase in the selected group.

At a curing age of 3 days, the M30-CH-1d group demonstrated optimal AfT production features. AfT crystals, characterized by their needle-like morphology, efficiently fill capillary pores and densify the matrix and enhance the early strength of the specimen [49]. The comparison of several alkali activation systems indicated that the M30-NH-10d group and the M30-NC-10d group exhibited distinct, distinctive peaks of calcite [50]. The development

of this inert phase will prompt Ca^{2+} ions and free SiO_2 to react with $\text{Al}(\text{OH})_4^-$ rapidly, hence impeding the hydration reaction.

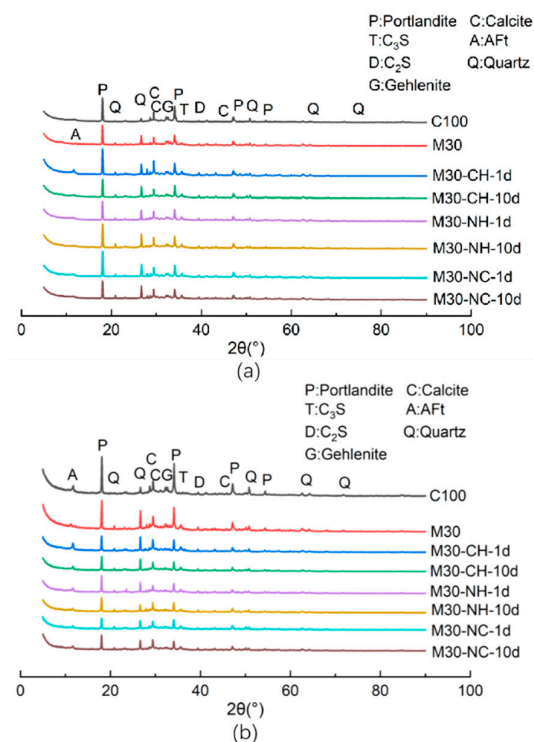


Figure 6. XRD analysis results of selected specimens after (a) 3-day curing age and (b) 28-day curing age.

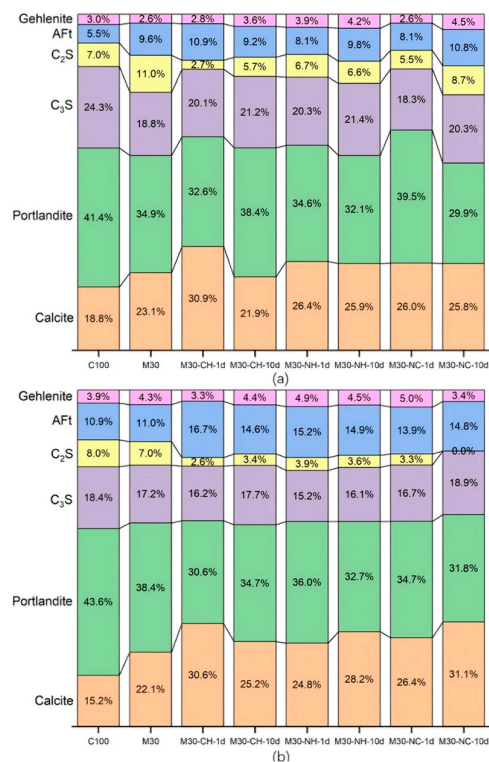


Figure 7. Ratios of each mineral phase content within selected specimens after (a) 3-day curing age and (b) 28-day curing age.

After 28 days of curing, the mineral phase composition of each specimen exhibited notable changes. The M30-CH-1d group exhibited the superior ability to generate AFt, indicating that CH-1d-IBA can facilitate AFt production and augment cement strength. Nonetheless, when the immersion duration in the $\text{Ca}(\text{OH})_2$ solution was prolonged to 10 days, the relative concentration of AFt diminished. The prolonged alkali treatment period may have resulted in the premature formation of a dense C-S-H gel layer, obstructing the subsequent hydration reaction. The M30-NH group had a more pronounced distinctive peak of gehlenite. The elevated Na^+ ion concentration of IBA following NaOH activation facilitates the production of N-A-S-H gel [51] with reduced strength and hinders the development of C-S-H, correlating with the observed decrease in macroscopic specimen strength.

Analysis of the activation mechanisms reveals that $\text{Ca}(\text{OH})_2$ alkaline treatment offers a dual benefit: it promotes the heterogeneous nucleation of AFt crystals via the introduction of Ca^{2+} ions, while simultaneously enhancing the dissolution of Al_2O_3 from IBA in the alkaline environment, leading to the formation of an Al-H gel that acts as a key precursor for AFt formation. Both short (1 day) and long (10 days) activation durations support the steady development of AFt. The underlying mechanism is attributed to an optimal alkaline treatment duration, which improves the reactivity of IBA while preventing the premature formation of a product layer that could act as a diffusion barrier. Quantitative data indicate that the AFt content in the M30-CH-1d group is 2.1% higher than that in the M30-CH-10d group. This optimized phase composition is a critical factor contributing to the significantly enhanced mechanical properties observed in the M30-CH-1d group.

3.4. TG/DTG

Figure 8 illustrates the TG/DTG curves of the specimens from each group cured for 3 and 28 days. The comparison indicates that various alkaline solutions and pretreatment durations significantly influence the hydration process of IBA-based mortar.

Among the specimens cured for 3 days, the M30-CH-1d group exhibited the highest weight loss rate of 3.83% during the S1 stage ($\sim 0\text{--}150^\circ\text{C}$), surpassing the M30-NH-1d group (3.44%) by 11.3% and the M30-NC-1d group (3.25%) by 17.8%. This suggests that the $\text{Ca}(\text{OH})_2$ solution efficiently facilitates the initial development of the C-S-H gel by addressing the Ca^{2+} shortage in IBA [46]. Nevertheless, the S1 weight loss rate of the M30-CH-10d group diminished to 3.45%, indicating that an excessively prolonged alkaline pretreatment environment leads to the premature formation of a dense C-S-H gel structure, hence impeding the further hydration of unreacted particles [52]. This effect was also observed in the M30-NH-10d group and the M30-NC-10d group, where the S1 weight loss rates diminished to 2.73% and 2.85%, respectively, representing reductions of 15.7% and 21.9% compared to the M30-NH-1d group and the M30-NC-1d group, respectively. During the S3 phase at $400\text{--}600^\circ\text{C}$, the M30-CH-1d group exhibited a weight loss rate of 2.42%, surpassing that of the M30-NC-1d group at 2.34% and the M30-NH-1d group at 2.17%. The XRD test results also demonstrate that CO_3^{2-} in the Na_2CO_3 alkaline solution combines with Ca^{2+} to produce CaCO_3 precipitate, hence depleting a portion of the active calcium supply and diminishing the stability of the hydration product. The S3 weight loss rate of the M30-NH-10d group ($\sim 2.04\%$) is lower than that of the M30-NH-1d group, suggesting that the elevated alkaline conditions of NaOH induce excessive depolymerization of the silicate network, dissociating Ca^{2+} and OH^- and facilitating the presence of $\text{Ca}(\text{OH})_2$ in an amorphous state [53].

Upon reaching 28 days of curing, the mass loss characteristics of each experimental group exhibit a novel evolutionary pattern. The S1 weight loss rate of the M30-CH-1d group rose from 3.83% to 5.02%, whereas the M30-NC-10d group experienced a modest increase

from 3.25% to 4.62%, demonstrating the substantial long-term carbonization effect of the Na_2CO_3 system [54]. In the S4 stage ($\sim 600\text{--}1000^\circ\text{C}$), the M30-NC-10d group exhibited the greatest weight reduction, totaling 2.85%. This indicates that the injection of CO_3^{2-} amplifies the carbonation reaction within the cement matrix, resulting in an unstable CaCO_3 phase. Conversely, the M30-CH group retained its superiority following 28 days of cure. The M30-CH-1d group attained the greatest weight loss during the S3 phase, illustrating its capacity to consistently supply an effective calcium source to facilitate hydration processes.

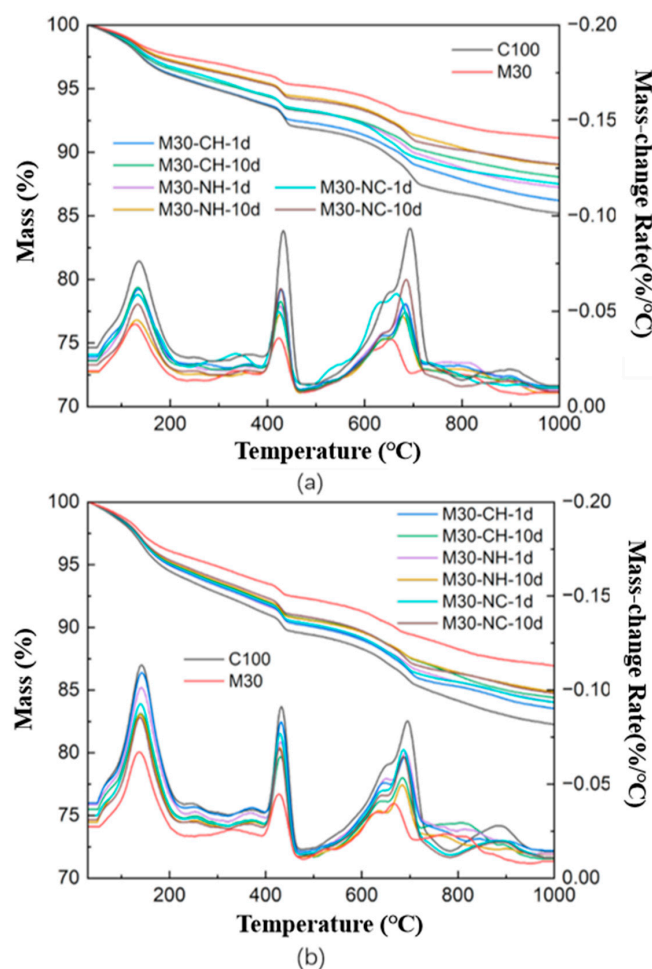


Figure 8. TG/DTG curves of selected specimens after (a) 3-day curing period and (b) 28-day curing period.

The comparison of various alkali treatment durations indicated that 1-day short-term alkali activation was predominantly more effective than 10-day long-term alkali activation. In the M30-CH group, the total weight loss rate for the M30-CH-1d group during the S1 and S3 stages was 6.25%, surpassing the 5.53% observed in the M30-CH-10d group. The disparity arises from the two-stage competition mechanism: In the short-term immersion phase, the alkaline environment expedites the dissolution of Al/Si active components in IBA and fosters the formation of C-(A)-S-H gel [55]. Conversely, in the long-term immersion phase, the increase in solution pH induces excessive depolymerization of silicates, resulting in the formation of a product layer that obstructs the interaction between water and unreacted particles. Furthermore, the M30-NC group exhibited systematic attenuation after 28 days of curing, which is strongly associated with the adverse consequences of the ongoing dissolution of Na^+ disrupting the gel charge equilibrium and CaCO_3 precipitation [54]. The $\text{Ca}(\text{OH})_2$ solution demonstrated the most effective activation of IBA after a one-day immersion, markedly enhancing the quantity of hydration products produced

by dynamic Ca^{2+} replenishment. Nonetheless, the Na_2CO_3 system necessitated control of treatment duration to mitigate the risk of carbonation and prevent prolonged deterioration of material characteristics.

3.5. FTIR

Figure 9 illustrates the FTIR spectra for each specimen group following 28 days of cure. The variations in distinctive peaks at 1466 cm^{-1} (C-O vibration), 1095 cm^{-1} (Si-O-Si vibration), 874 cm^{-1} (CO_3^{2-} bending vibration), and 425 cm^{-1} (M-O vibration) indicate the influence of distinct alkali treatments on the regulation of hydration products.

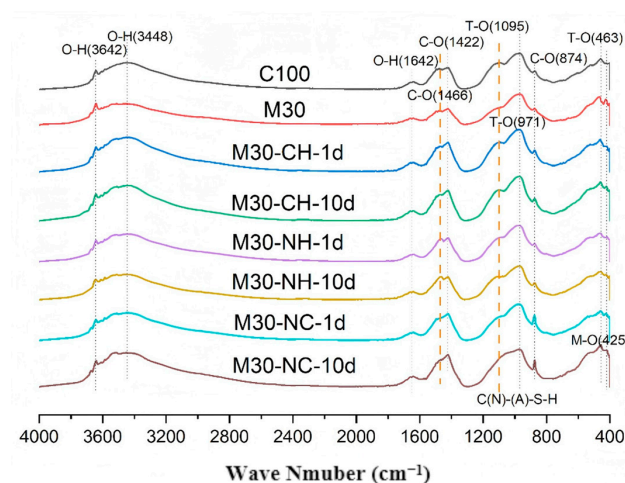


Figure 9. FTIR spectra of specimens in selected groups after 28 days of curing.

The M30-NC-1d and M30-NC-10d groups have a pronounced absorption peak at 874 cm^{-1} , signifying that the complexation process of CO_3^{2-} with metal ions in IBA results in carbonate enrichment, hence impeding the production of aluminosilicate gel (the peak intensity at 1095 cm^{-1} is diminished). The M30-CH-1d and M30-CH-10d groups exhibit a notable enhancement in peak intensity at 1095 cm^{-1} , alongside a reduction in the metal oxide peak at 425 cm^{-1} , thereby substantiating that Ca^{2+} significantly facilitates silicate depolymerization and gel deposition, culminating in optimal densification outcomes. The M30-NH-1d and M30-NH-10d groups exhibit moderate peak intensity at 1095 cm^{-1} , accompanied by a faint carbonate peak at 1466 cm^{-1} , indicating that sodium ion movement interferes with the systematic arrangement of the gel.

3.6. Microstructures

Figure 10 shows the microstructures of IBA with different alkali types of pretreatments. The images indicate that the amorphous phase in the activated IBA was partially dissolved, which is in line with a study [49], resulting in the formation of an amorphous lamellar structure and flocculent C-S-H gel on the surface.

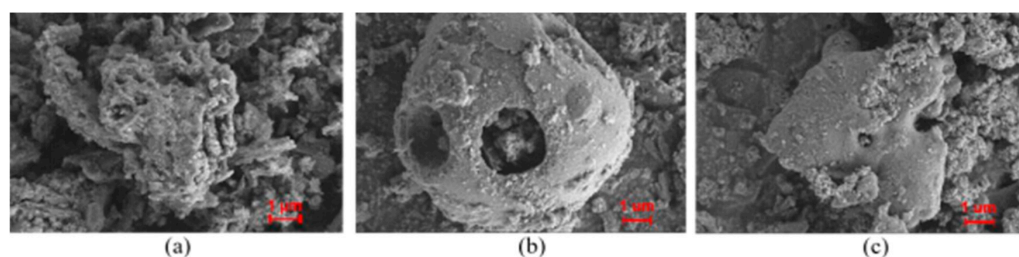


Figure 10. Microstructures of IBA: (a) CH-1d; (b) NH-1d; and (c) NC-1d.

Figure 11 shows the microstructures of selected specimens cured for 28 days. The microstructures of the M30-CH-1d group are more compacted compared to those of the other groups. After 28 days of curing, the specimen surface displays a characteristic composite structure of hydration products: many needle-like ettringite (AFt) interlaced with flocculent C-S-H gel, interspersed with flake-like $\text{Ca}(\text{OH})_2$ crystals, creating a dense three-dimensional network. The radial expansion of ettringite and the persistent encapsulation of the C-S-H gel substantially augment the material's overall strength. In the M30-CH-10d group, the quantity of ettringite and $\text{Ca}(\text{OH})_2$ crystals diminishes, whilst the C-S-H gel layer increases in thickness and achieves a more uniform distribution. Microcracks and pores are seen in certain regions. This indicates that prolonged alkali treatment results in excessive densification of the C-S-H gel, hence impeding the ongoing breakdown of $\text{Ca}(\text{OH})_2$ and subsequent hydration processes.

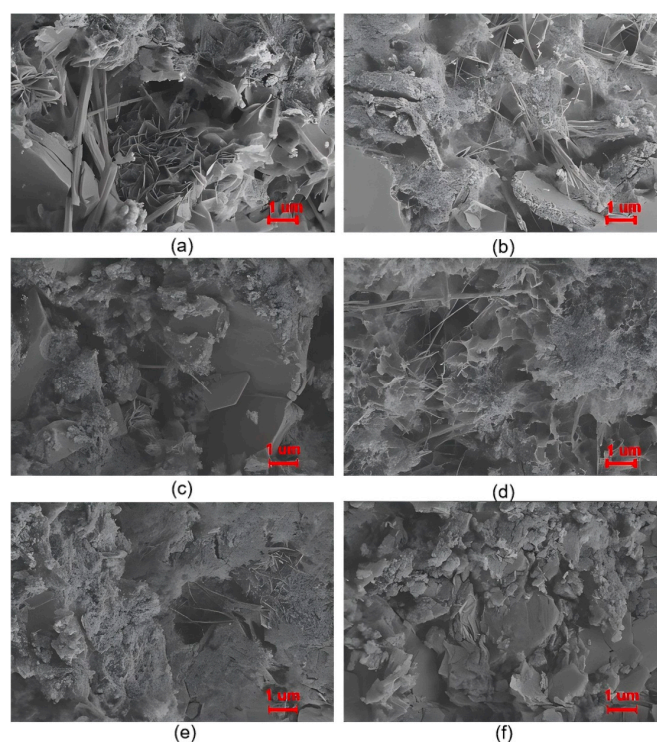


Figure 11. Microstructures of the groups after 28-day curing: (a) M30-CH-1d group; (b) M30-CH-10d group; (c) M30-NH-1d group; (d) M30-NH-10d group; (e) M30-NC-1d group; and (f) M30-NC-10d group.

In the M30-NH-1d group, the C-S-H gel has a mostly loose flocculent structure, characterized by diminutive ettringite crystals and sparsely arranged $\text{Ca}(\text{OH})_2$ platelets. The porous structure indicates that the extremely alkaline NaOH environment accelerates the depolymerization of the silicate network, obstructing the stable crystallization of the hydration products. The microstructure of the M30-NH-10d group progressively deteriorates, characterized by localized agglomeration of the C-S-H gel and the near-absence of ettringite, supplanted by chaotic accumulations of amorphous products. This structural defect indicates that although a robust alkaline environment can temporarily activate the silica–alumina phase, prolonged exposure results in the dissolution of active components and the carbonization of certain hydration products, culminating in the formation of non-gelling sodium salts that diminish matrix density.

Microstructural analysis also shows that the M30-NC-1d group exhibits a distinctive layered-granular composite structure, wherein the C-S-H gel coexists with calcite (CaCO_3) microcrystals, accompanied by negligible ettringite presence. The production of CaCO_3

depletes Ca^{2+} , hence affirming the detrimental effect of carbonization on material stability. The microstructure of the M30-NC-10d group indicates enhanced aggregation of CaCO_3 particles, fracturing of the C-S-H gel network into discrete regions, and a notable loosening of the structure. These morphological characteristics align with the diminished mechanical strength relative to the M30-NC-1d group, suggesting that prolonged Na_2CO_3 treatment intensifies the development of carbonization products and significantly impedes the synthesis of efficient hydration products.

In conclusion, microstructural analyses show that the M30-CH-1d group markedly enhances the material's densification and mechanical strength by facilitating the synchronized development of AFt, C-S-H gel, and $\text{Ca}(\text{OH})_2$ crystals. Conversely, prolonged exposure to an alkaline environment or Na_2CO_3 activation treatment promotes microcrack propagation and the development of carbonation products. These structural faults directly result in a reduction in the material's mechanical characteristics.

3.7. MIP and X-CT Analysis

This experiment employed mercury intrusion porosimetry (MIP) to evaluate the porosity parameters of specimens cured for 3 and 28 days. As shown in Figure 12, the pore size distribution curves and cumulative pore volume curves for specimens cured for 3 and 28 days were utilized to comprehensively analyze the impacts of alkali solution type and alkali treatment duration on the porosity properties of IBA-modified mortar. From the appearance of the test block and the X-CT test results, it can be seen that under alkaline conditions, the metal aluminum in IBA reacts to generate hydrogen gas bubbles. Due to the much lower density of bubbles compared to the slurry, they will float upwards and gather at the top of the test block before setting [5].

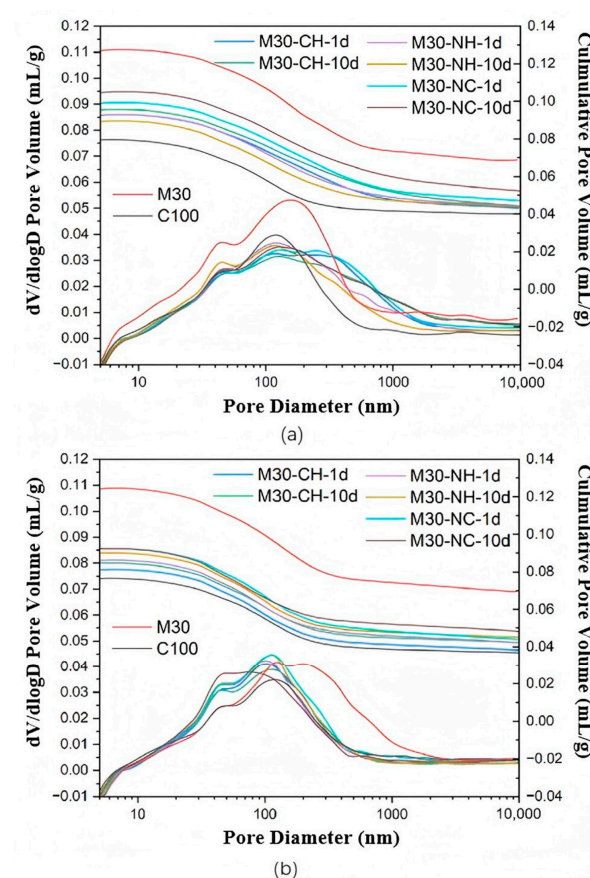


Figure 12. Pore size distribution of specimens after being cured for (a) 3 days and (b) 28 days.

These findings indicate that the evolution of pore structure in IBA mortar specimens is significantly influenced by both time and solution conditions. At 3 days of age, the porosity of the M30-NH-10d group was surpassed only by that of the C100 group and was lower than that of the M30-NH-1d group within the same system. This phenomenon arises from the ongoing depolymerization of aluminosilicates in the IBA due to the strong alkalinity of the NaOH solution: increased dissolution and release of amorphous silicate and alumina components in the NH-10d-IBA occur, resulting in gel products during early hydration that effectively occupy some of the original pores. Nonetheless, a considerable quantity of macropores over 100 nm in diameter remained present across all experimental groups. This is mainly attributable to the insufficient development of hydration products during the initial activation phase, which constrains pore-filling efficacy.

At 28 days, the M30-CH group had superior pore refinement capabilities: the M30-CH-1d subgroup recorded the lowest porosity (~16.28%) and the smallest average pore width (~13.4 nm), highlighting the efficacy of the calcium ion migration–deposition mechanism. Extended immersion duration yielded minimal enhancement in porosity, suggesting that the $\text{Ca}(\text{OH})_2$ activation process approached saturation within one day. The M30-NH group demonstrated a dual temporal effect: a 10-day immersion expedited pore filling initially, but the inhibition of sodium ion migration during prolonged hydration resulted in a reduction in gel packing density, ultimately leading to the porosity of M30-NH-10d exceeding that of M30-NH-1d.

The M30-NC group exhibited elevated porosity during the curing period, attributed to the complex reaction between CO_3^{2-} and metal ions in IBA. Carbonate ions preferentially interacted with calcium, aluminum, and other ions to form soluble complexes, thereby postponing the deposition of the gel product.

X-ray computed tomography (X-CT) was employed to analyze the multiscale pore structure of 28-day-old specimens, addressing the limitations of mercury intrusion porosimetry in three-dimensional pore characterization. The integration of three-dimensional reconstruction (Figure 13), two-dimensional cross-sectional images (Figure 14), and grayscale distribution characteristics (Figure 15) systematically elucidates the modulation of the pore structure of IBA-modified mortars by various alkali activation pretreatments.

The three-dimensional reconstruction results indicate that the pore distribution in the M30-NC-1d group demonstrates considerable connectivity and directionality. A significant quantity of horizontally oriented fracture-like pores is localized in the anterior quarter of the specimen's cross-section. This pertains directly to the complexation of aluminum in IBA by CO_3^{2-} . In the Na_2CO_3 activation system, CO_3^{2-} interacts with Al^{3+} to create a stable, soluble complex, thereby inhibiting aluminum ions from engaging in the hydration reaction that produces aluminosilicate gel. The lack of gel products leads to an absence of cementing material on the pore walls, causing the empty pores to gradually grow and interpenetrate under stress [56]. Furthermore, the complex may undergo hydrolysis during subsequent curing, resulting in the release of Al^{3+} , which combines with OH^- in the alkaline milieu to produce $\text{Al}(\text{OH})_3$ colloid, leading to localized volume expansion [57], hence intensifying the shear failure of the pore walls.

Two-dimensional CT scans further validate the disparities in pore management among various alkali treatments. In the M30-CH-1d group, the pores are predominantly isolated gelled pores characterized by smooth edges and a close integration with the matrix. The pores are predominantly located in regions abundant in hydration products, suggesting that the C-S-H gel generated by calcium ion migration efficiently occupies the interfacial transition pores, hence enhancing matrix continuity considerably. Conversely, capillary and macroscopic pores are present in the M30-NH-1d group, indicating that increased

sodium ion migration results in localized gel stacking disintegration, hence diminishing pore healing efficacy. In the M30-NC-1d group, the pores demonstrate a chain-like interconnected structure, with certain regions creating continuous fracture zones, corroborating the three-dimensional reconstruction findings and suggesting the lack of pore self-repair mechanisms within the carbonate system.

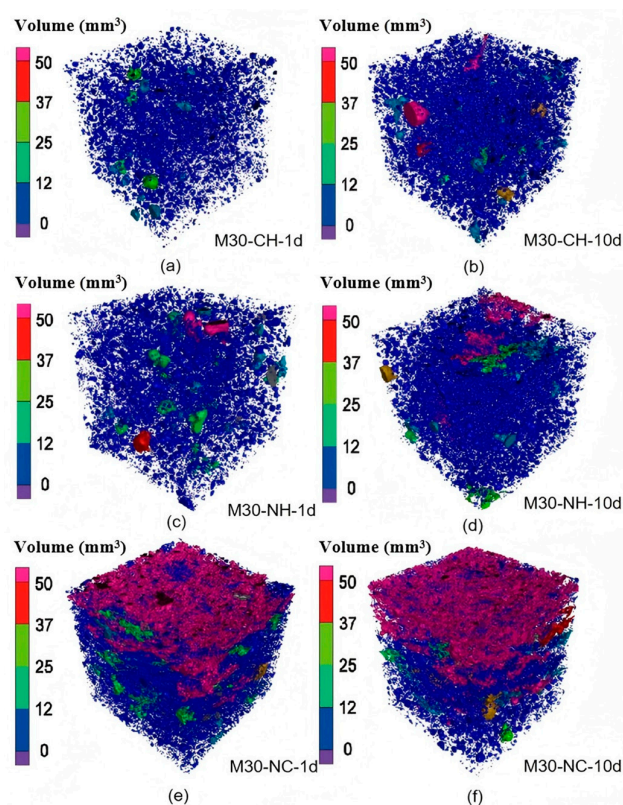


Figure 13. Three-dimensional CT reconstruction results of selected groups: (a) M30-CH-1d, (b) M30-CH-10d, (c) M30-NH-1d, (d) M30-NH-10d, (e) M30-NC-1d and (f) M30-NC-10d at 28 days of age.

The properties of grayscale distribution (Figure 15) establish a dynamic link with the hydration process. The grayscale curve of the M30-CH-1d group has a tight unimodal distribution, signifying an increase in hydration products and a reduction in pore area. Moreover, the resultant hydration products exhibit a more uniform densification, illustrating that $\text{Ca}(\text{OH})_2$ alkaline treatment can efficiently enhance the gelling characteristics of IBA. Conversely, M30-NC-1d displays the least hydration products and the most extensive pore area among the pretreated chemically activated groups, highlighting the inadequacies of Na_2CO_3 alkaline treatment in enhancing the gelling capabilities of IBA.

The combined application of MIP and X-CT provides a comprehensive, multiscale understanding of pore structure evolution in IBA-modified mortars. The correspondence between the two techniques is evident in several key aspects. The lower total porosity measured by MIP for the M30-CH-1d group ($\sim 16.28\%$) aligns directly with the X-CT observations of a denser matrix dominated by isolated, gel-like pores (Figure 14a) and a tighter grayscale distribution (Figure 15). Conversely, the higher porosity and greater volume of large capillaries (>100 nm) indicated by MIP for the Na_2CO_3 -treated groups (M30-NC) correlate with the X-CT findings of interconnected, fracture-like pore networks (Figures 13 and 14c). This synergy confirms that the superior mechanical performance of the $\text{Ca}(\text{OH})_2$ -activated specimens stems from effective pore refinement and isolation, while the inferior performance of the Na_2CO_3 -activated ones is linked to the development of connected macro-porosity.

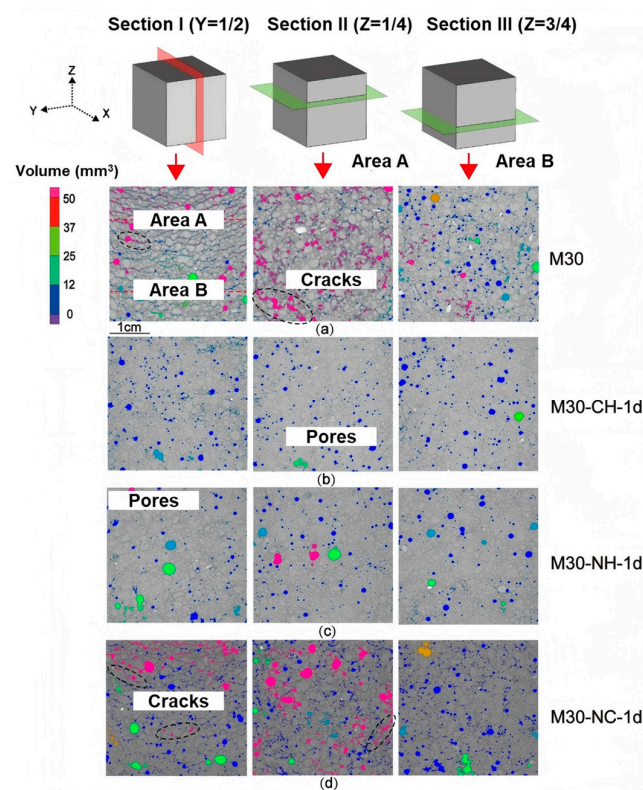


Figure 14. 28-day 2D CT image analysis of selected groups: (a) M30, (b) M30-CH-1d, (c) M30-NH-1d and (d) M30-NC-1d.

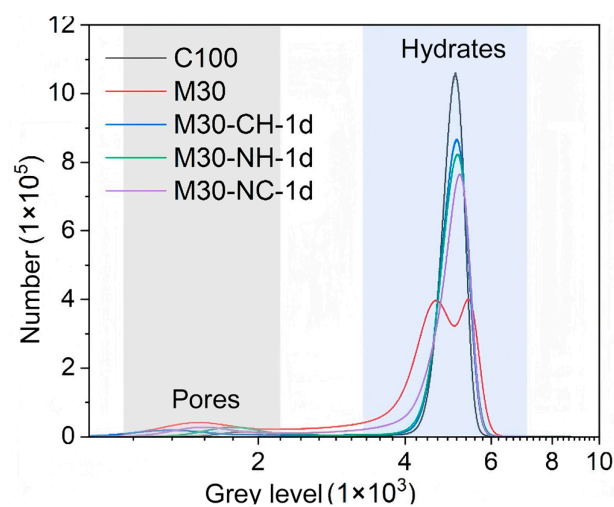


Figure 15. Grayscale distribution of selected specimens after CT scans.

It is important to acknowledge the inherent limitations of each technique to properly interpret the data. MIP, while excellent for quantifying pore size distribution and total intrudable porosity, is subject to the “ink-bottle” effect, where the measured pore throat size may not represent the actual pore body size, potentially underestimating the volume of large but narrowly connected pores [58,59]. Furthermore, MIP may not fully capture pores that are only accessible through very small entrances or that are closed within the sample volume. On the other hand, X-CT excels in visualizing three-dimensional pore morphology, connectivity, and distribution but has a practical resolution limit ($\sim 1\text{--}2\ \mu\text{m}$ in this study), making it insensitive to nano-scale pores within the C-S-H gel that are crucial for durability [60]. The quantification of porosity from X-CT is also dependent

on the selection of grayscale thresholds, which can introduce subjectivity. Therefore, the MIP and X-CT results are best interpreted as complementary: MIP provides quantitative metrics for the finer pore regime, while X-CT offers qualitative and spatial context for the larger, structurally significant pores. This integrated approach overcomes the individual limitations and provides a robust characterization of the complex pore systems developed under different activation regimes [5].

3.8. Carbon Footprint

The cradle-to-gate carbon footprint results (Figure 16a) show clear differences by activator type: NC-IBA (Na_2CO_3 -treated IBA) has the highest footprint (0.1250 kg CO_2 per functional unit), followed by CH-IBA ($\text{Ca}(\text{OH})_2$) at 0.0727 kg CO_2 and NH-IBA (NaOH) at 0.0557 kg CO_2 . Two causes explain this ordering. First, at equal molar dosing, the mass of the reagent differs markedly ($\text{Na}_2\text{CO}_3 \approx 106.0 \text{ g}\cdot\text{mol}^{-1} > \text{Ca}(\text{OH})_2 \approx 74.1 \text{ g}\cdot\text{mol}^{-1} > \text{NaOH} \approx 40.0 \text{ g}\cdot\text{mol}^{-1}$), so the same molar treatment requires more mass and therefore more embodied emissions. Second, the production of Na_2CO_3 (typically via the Solvay process or natural soda ash calcination) is considerably more energy-intensive than that of $\text{Ca}(\text{OH})_2$ or NaOH , owing to high-temperature reactions (up to 900°C) and the associated CO_2 release from limestone decomposition [61,62]. These combined effects make the Na_2CO_3 -based pretreatment pathway more carbon-intensive than $\text{Ca}(\text{OH})_2$ or NaOH activation routes.

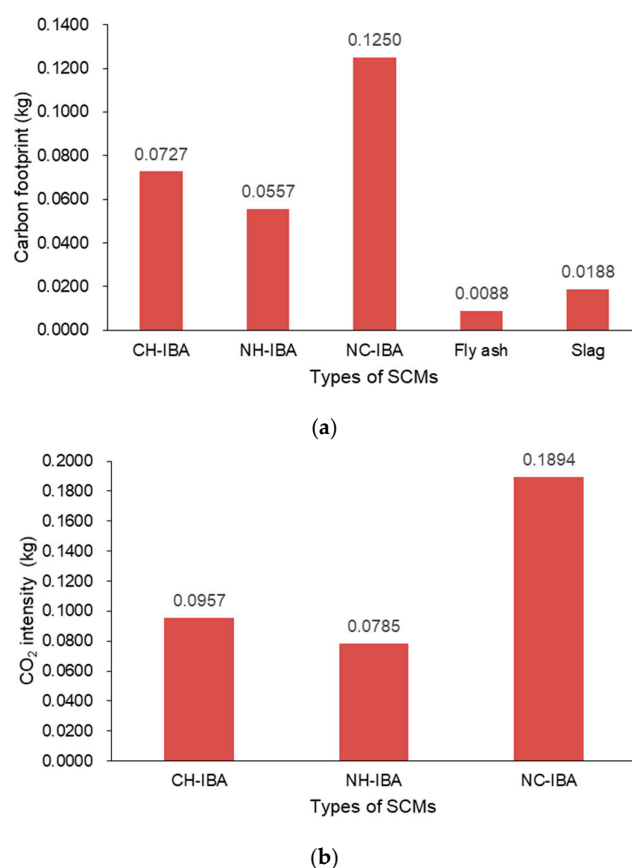


Figure 16. (a) Carbon footprint and (b) CO_2 intensity associated with the production of 1 kg SCMs using the impact method CML2000.

When these chemically treated IBA footprints are compared with conventional industrial SCMs, the contrast is stark: fly ash ($\sim 0.0088 \text{ kg CO}_2$) and blast-furnace slag ($\sim 0.0188 \text{ kg CO}_2$) are an order of magnitude (or more) lower than even the lowest activator-treated IBA case. Fly ash and GGBS are industrial by-products whose upstream burdens are mostly

allocated to the power or steel production systems, so their incremental cradle-to-gate CO₂ for use as SCMs is very small and typically limited to collection, classification, and grinding [43,63]. In contrast, the chemically treated IBA cases reported here include the embodied emissions of the chemical reagents used for activation, and those reagents are carbon-intensive on a per-kilogram basis. Emission factors for common activators show substantial CO₂ intensities, so applying them at equal molar doses increases the IBA pathway's total emissions markedly. Moreover, many current IBA valorization workflows in the literature rely primarily on mechanical activation (ball milling and drying) [64,65] rather than chemical pretreatment; because milling/drying consumes energy but does not add reagent-embodied CO₂, mechanically activated IBA can have a much lower marginal footprint than chemically treated IBA if only those steps are used.

When the cradle-to-gate emissions of chemically pretreated IBA were normalized by their measured reactivity, the ranking of activation routes did not change too much, as shown in Figure 16b. NaOH treatment (NH-IBA) is the most carbon-efficient per unit of measured reactivity. Although CH-IBA shows the highest absolute activity (76%), NaOH's lower reagent mass per mole and comparatively lower embodied emissions produce the lowest CO₂ per unit activity.

Assumptions and Uncertainties: Several assumptions underlie this assessment, contributing to uncertainty. The life-cycle inventory data for alkali production were sourced from the Ecoinvent v3.8 database, which represents European industrial averages. Their application to a Chinese context may introduce uncertainty due to differences in the grid electricity mix and production technologies. The exclusion of transportation for raw IBA and water use simplifies the model but has a negligible impact on the comparative results, as these steps are common to all pretreatment options. The most significant assumption is using the 28-day strength activity index (H₂₈) as the sole proxy for the functional performance (reactivity) of the SCM. While H₂₈ is a standard metric, it encapsulates only the mechanical contribution, omitting potential durability effects. Furthermore, the H₂₈ values for fly ash and slag are based on typical ranges from the literature [40,57], which may vary. Despite these uncertainties, the primary goal of this screening-level LCA is to provide a consistent comparative assessment of the relative carbon efficiency of different activation routes for IBA, rather than absolute footprint values.

Comparison Under Equivalent Performance Criteria: The comparison in Figure 16a on a per-mass basis highlights the fundamental carbon burden of chemical activation. However, a more functionally equitable comparison must account for the material's effectiveness in cement replacement. This is achieved through the reactivity-normalized CO₂ intensity ($C_i = C_0/H_{28}$), presented in Figure 16b. This metric answers the question: "How many kg of CO₂ are emitted to achieve one unit of reactivity gain?". While conventional SCMs (fly ash and slag) have very low cradle-to-gate emissions per kg (Figure 16a), their normalized C_i must be evaluated based on their own typical H₂₈ values. For instance, a fly ash with H₂₈ of 80% would have a C_i of approximately 0.011 kg CO₂ per % reactivity, which remains significantly lower than the chemically treated IBA options. This analysis underscores that although chemical pretreatment can enhance IBA's reactivity substantially (e.g., H₂₈ of 76% for CH-IBA), the associated carbon cost of the reagents currently prevents it from matching the carbon efficiency of established industrial by-product SCMs on a performance-adjusted basis. The value of pretreatment lies in upgrading a locally abundant, low-value waste into a viable SCM, potentially reducing landfilling and virgin material use within a regional circular economy context, even if its carbon intensity per unit performance is higher.

The reactivity-normalized metric makes carbon footprint performance tradeoffs explicit, but it should be interpreted alongside other constraints: hydrogen generation risk (metallic Al), durability, leachability of heavy metals, process cost, and scale-up feasibility.

ity. For example, NaOH may give the best CO₂/reactivity but can increase H₂ evolution and ASR risk if not carefully controlled. Ca(OH)₂ often offers better compatibility with cement matrices and lower H₂ risk, which may justify a modestly higher normalized CO₂ in some applications.

4. Conclusions and Outlook

This study systematically evaluated chemical pretreatment as a method to enhance the cementitious properties of municipal solid waste incineration bottom ash (IBA). By comparing three alkaline activators (Ca(OH)₂, NaOH, and Na₂CO₃) across a range of treatment durations (1–10 days) and integrating performance assessment with microstructural and environmental analysis, the following key conclusions are drawn:

(1) Ca(OH)₂ pretreatment for 1 day emerged as the optimal strategy, achieving a 28-day strength activity index (H₂₈) of 76%. This treatment efficiently supplements Ca²⁺ to promote the nucleation of Aft and the C-S-H gel while enhancing Al₂O₃ dissolution, leading to a dense microstructure with the lowest porosity and the best mechanical performance. Prolonged treatment (>3 days) caused premature gel densification, reducing effectiveness.

(2) Sodium-based activators showed distinct limitations. NaOH introduction of Na⁺ disrupted gel charge balance, while Na₂CO₃ induced significant carbonation, both leading to higher porosity and inferior strength. Extended treatment with either activator exacerbated these issues, underscoring the importance of controlled activation time.

(3) Treatment duration exhibited a clear non-linear optimum. A short (1-day) treatment effectively activated surface phases without causing detrimental over-dissolution or passivation. Longer treatments uniformly reduced performance, regardless of the alkali type, highlighting that activation is kinetically limited rather than diffusion-controlled under these conditions.

(4) From an environmental perspective, although NaOH showed the lowest CO₂ intensity per unit of reactivity gained, Ca(OH)₂ offered the most favorable overall balance, providing strong activation with moderate carbon emissions and better compatibility with cement chemistry, making it a practical and sustainable choice for IBA valorization.

(5) This work demonstrates a viable pathway to transform IBA into a functional supplementary cementitious material. The recommended protocol—treatment with 0.2 mol/L Ca(OH)₂ for 1 day—significantly enhances reactivity while maintaining a manageable carbon footprint, supporting the circular economy in construction.

Outlook: This study focused on chemical activation under laboratory conditions. Future work should address: (i) the long-term durability and leaching behavior of IBA-incorporated concrete, (ii) the economic feasibility and scaling-up of the pretreatment process, and (iii) the potential use of blended or low-concentration alkaline solutions to further reduce cost and environmental impact. Additionally, the interaction between pretreated IBA and different cement types warrants further investigation to broaden applicability.

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