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Microstructure and Nanomechanical Characteristics of Hardened Cement Paste Containing High-Volume Desert Sand Powder

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Abstract: Desert areas contain abundant desert sand (DS) resources, and high-volume recycling of DS resources as components of cement-based materials can achieve high-value applications. In this paper, DS was processed into desert sand powder (DSP) and replaced with cement in high volumes (20 wt.%–60 wt.%) to produce cement pastes. The mechanical properties, heat evolution, nanomechanical characteristics, microstructure, and economic and environmental impact of cement pastes were studied. The results show that adding 20 wt.% DSP increases the compressive strength of pastes and accelerates cement hydration, compared with the control group (0 wt.% DSP). Meanwhile, incorporating an appropriate amount of DSP (20 wt.%) effectively reduces porosity, increases the proportion of harmless and less harmful pores, and reduces the proportion of more harmful pores. From the perspective of nanoscopic properties, the addition of 20 wt.% DSP increases the C-S-H volume fraction, especially enhancing the transformation of low-density C-S-H to high-density C-S-H. Notably, the sample incorporating 60 wt.% DSP exhibits the lowest values for CI coefficients (13.02 kg/MPa·m³) and Cp coefficients (2.29 USD/MPa·m³), thereby validating the application of high-volume DSP feasibility in cement-based materials.

Keywords: desert sand powder; high volume; microstructure; nanomechanical properties; heat evolution; environmental impact



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1. Introduction

Concrete is extensively utilized in infrastructure construction owing to its affordable cost, diverse raw material sources, ease of molding, and exceptional mechanical attributes. Given the rapid urbanization across the globe and the never-ending improvement of human requirements, the need for concrete has increased sharply. Worldwide, 25 billion tons of concrete are manufactured annually, of which China and India account for roughly 11.5 billion tons [1]. This places higher demands on the production of raw materials such as cement, sand, and gravel. However, the extensive use of cement and over-exploitation of natural resources, such as sand and stone, has led to environmental degradation and has significantly reduced their reserves [2]. Meanwhile, the process of producing cement clinker generates a significant amount of CO₂ emissions, accounting for approximately 8% of global CO₂ emissions, showing that the excessive use of cement is not conducive to sustainable development [3]. Therefore, mankind must find a supplementary or alternative raw material to replace cement, sand, and gravel to alleviate the conflict between urban construction and environmental development.

Continents across the globe have abundant desert areas, and the Chinese desert area is 1.533 million km², and desertification continues at a rate of 1560 km² per year, aggravating the ecological problems caused by environmental pollution [4]. Desert areas contain abundant desert sand (DS) resources, and using DS materials as an alternative to components in cement-based materials can effectively mitigate environmental pollution

and curtail CO₂ emissions in the construction industry. The application of DS in the building industry has been widely studied. Elipse et al. [5] pointed out that the physical properties of DS were mainly characterized by low fineness modulus, smooth surface, regular shape, single gradation, and small particle size. Wang et al. [6] employed DS in the fabrication of 3D-printed concrete with different sand-to-binder ratios (1.25–1.90) and found that adding DS reduced the mixture fluidity and enhanced the freeze–thaw resistance in the concrete. Meanwhile, when the sand-to-binder ratio was 1.7, the compressive strength of concrete was the highest (41.2 MPa). Yan et al. [7] utilized DS to replace fine aggregate, and found that the compressive strength of concrete decreased as the DS substitution level increased. Compared with river sand, apart from SiO₂, DS contained more alkaline oxides such as CaO, Al₂O₃, Fe₂O₃, MgO, K₂O, and Na₂O, indicating that DS may have pozzolanic activity [8]. Meanwhile, the material pozzolanic activity increased as the particle size decreased. Therefore, some scholars consider preparing DS as desert sand powder (DSP) for application in the construction industry [9,10]. Guettala and Mezghiche [8] proved that DSP with a fineness measurement of 4000 cm²/g exhibited pozzolanic activity, and found that adding 5 wt.%–10 wt.% DSP can enhance the 28-day compressive strength in the specimen. Furthermore, the 28-day compressive strength of specimens with 20 wt.% DSP was almost the same as that of the sample without DSP. Alhozaimy et al. [11] studied the strength changes of composite cement with DSP under autoclaved curing, and found that the compressive strength increased as the DSP substitution level increased. Zaitri et al. [12] employed experimental and simulation methods to study the DSP effect on mortar, and found that the addition of DSP was beneficial to the compressive strength and fluidity of the specimen. In addition, the use of DSP in building materials can significantly reduce production costs, and is beneficial to the sustainability and economy of construction [6,9,10]. For example, replacing cement with 10 wt.% DSP to prepare cement paste can reduce CO₂ emissions by about 9% [11]. Therefore, the utilization of DSP as a component in the building industry is advantageous in minimizing CO₂ emissions, while also effectively promoting the recycling of desert resources.

Previous studies have indicated the viability of DSP's ability to serve as supplementary cementitious materials (SCMs). However, the influence of DSP on cement-based materials' properties was not determined. Meanwhile, the DSP application was limited to the low substitution level. Therefore, this article employed high-volume (20 wt.%–60 wt.%) DSP to produce cement paste and explored the DSP effect on the mechanical properties, microstructure, and economic and environmental impacts of cement pastes. In addition, nanoindentation technology was used to explore the changes in its nanoscopic properties and the action mechanism of DSP to provide theoretical support for the subsequent application of high-volume DSP in engineering.

2. Materials and Methods

2.1. Materials

Type I Portland 42.5 cement (PC) and DSP were employed as binders, of which PC was produced by Shaoxing Zhaoshan Building Materials Co., Ltd., Shaoxing, China, and DS came from the Maowusu Desert in China. The DS was pulverized in a ball mill for 30 min and was subsequently filtered through a 75 µm sieve to acquire DSP with a particle size not exceeding 75 µm. The median particle sizes of PC and DSP are 15.2 µm and 13.4 µm, respectively, tested using a mastersize-3000 laser particle size analyzer (Malvern, UK, Malvern Panalytical), as depicted in Figure 1. The main oxide components of PC and DSP were analyzed using X-ray fluorescence spectrometry (XRF) (UK, Malvern Panalytical). PC mainly consists of CaO, while DSP is mainly composed of SiO₂ and Al₂O₃ and contains other basic oxides, as depicted in Table 1. The mixing water was tap water, and a polycarboxylate-based superplasticizer (SP) with a water-reducing capacity of 25% was added.

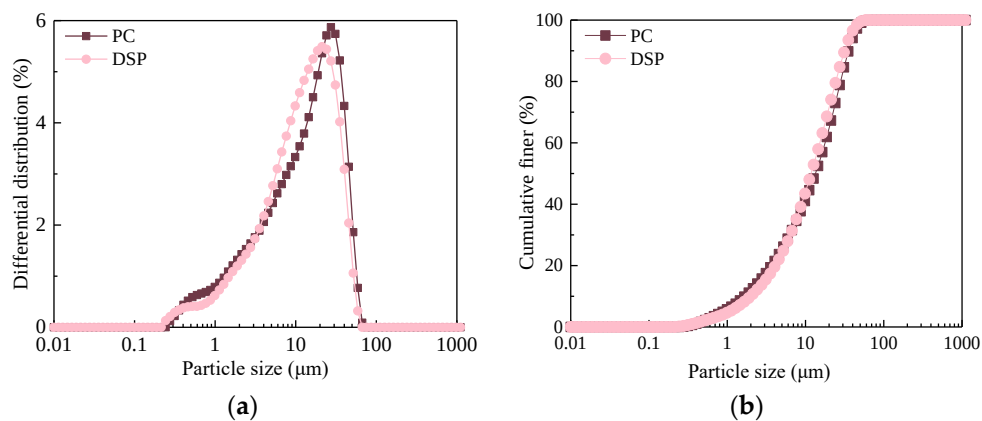


Figure 1. Particle size of binders, (a) distribution, and (b) cumulative curves.

Table 1. Chemical composition of binders (wt.%).

Materials	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	Loss on Ignition
PC	68.8	15.77	3.3	3.36	4.07	1.35	2.19
DSP	17.19	36.94	25.01	3.01	6.1	5.81	/

2.2. Mixing Proportions and Preparation

DSP was used to replace PC in preparing cement pastes, with 20 wt.%, 40 wt.%, and 60 wt.% DSP employed in this experiment. All mixtures were prepared with a constant water-to-binder ratio of 0.35 and 0.06 wt.% SP, as shown in Table 2. The stirred cement paste was cast into a mold with a size of 40 mm × 40 mm × 40 mm and cured at room temperature (20 °C) for 1 days. Following demolding, the specimens were transferred to a standard curing room (20 °C ± 2 °C, 95% ± 3% RH) for further curing. The detailed process can be seen in Figure 2.

Table 2. Mix design of cement pastes.

Groups	w/b	Mix Proportions (wt.%)		
		PC	DSP	SP
CP-0DSP	0.35	100	0	0.06
CP-20DSP		80	20	0.06
CP-40DSP		60	40	0.06
CP-60DSP		40	60	0.06

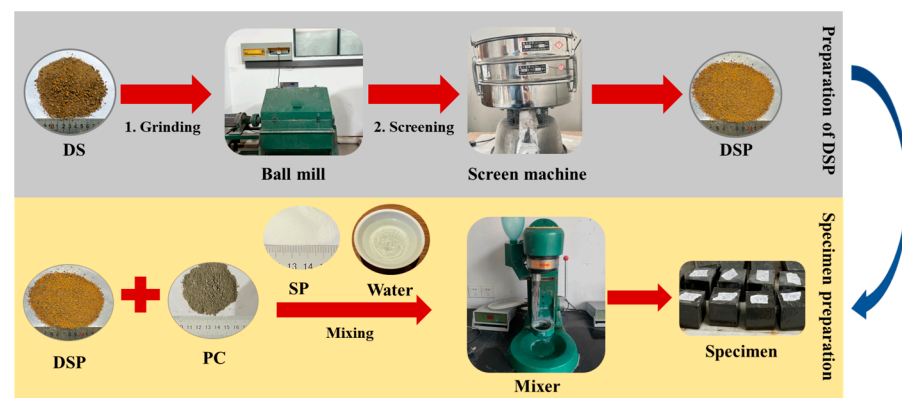


Figure 2. Preparation and characterization process of cement pastes with DSP.

2.3. Test Methods

2.3.1. Compressive Strength Test

The compressive strength of the cubic specimens was measured using a TENSON 600 kN servo-hydraulic testing machine (Shaoxing, China, Zhejiang Geotechnical Instrument Manufacturing Co., Ltd.) at 7 days, 28 days, and 60 days, according to Chinese standard GB/T 17671 [13]. The loading speed was set to 2400 ± 200 N/s. Data with an error exceeding 10% were discarded, and the mean value of the remaining data was considered to be the actual compressive strength. Each group used at least 6 samples for compressive strength testing.

2.3.2. Hydration Heat

Using a TAM Air isothermal calorimeter (New Castle, DE, USA, Thermometric AB), isothermal calorimetry measurements were conducted on cement pastes for 72 h, maintaining a temperature of 20 °C. The mixture was stirred externally before being placed in the calorimeter. The water–binder ratio was constant at 0.35, and approximately 3 g of binder was placed into the channel.

2.3.3. Microstructure

After 28 days, samples were broken into 1–4 mm pieces and soaked in isopropyl alcohol for 7 days to remove the water and terminate the hydration reaction. The sample was dried under a vacuum, and a thin gold layer was deposited on the samples with a vacuum sputtering coater to enhance their surface conductivity. Subsequently, the specimen micromorphology was observed using an SU3800 instrument (Tokyo, Japan, HITACHI) at an accelerating voltage of 10.0 kV. Meanwhile, the pore structure was analyzed with the mercury intrusion porosimetry (MIP) test using a Mercury Porosimeter (Norcross, GA, USA, Micromeritics) with an operating pressure of up to 340 MPa.

2.3.4. Nanoindentation

The 28-day samples were cut into 1 cm³ pieces and soaked in epoxy resin for curing, followed by grinding, polishing, and cleaning. After that, the vacuum-dried samples were measured with a Hysitron Ti Premier nanomechanical tester (Billerica, MA, USA, Bruker) to obtain their nanoscopic properties. The loading regime was configured as follows: 5 s of load application, followed by a 2 s holding period, and concluding with a 5 s unloading phase. The load was set at 4000 µN.

2.3.5. Environment and Cost Assessment

Due to DS being easily obtained from desert areas, the CO₂ emissions and cost of DSP were significantly lower than PC. The economic and environmental impacts of cement pastes containing 0 wt.%–60 wt.% DSP are evaluated. The CO₂ emissions and costs of raw materials are depicted in Table 3. In addition, the CO₂ emissions coefficient (CI, kg/MPa·m³) and cost coefficient (C_p, USD/MPa·m³) of cement paste under unit compressive strength are quantitatively calculated through Equations (1) and (2) [14].

$$C_p = \frac{Cost}{f_c} \quad (1)$$

$$CI = \frac{\text{embodied CO}_2 - e \text{ emission}}{f_c} \quad (2)$$

where Cost refers to the total cost in 1 m³ mixture (USD/m³), f_c denotes the 28-day compressive strength of cement pastes (MPa), and embodied CO₂-e emission represents the total weight of CO₂ in 1 m³ mixture (kg/m³).

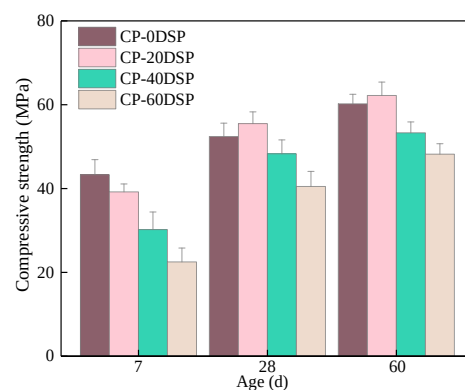
Table 3. The CO₂ emissions and costs of raw materials.

Materials	CO ₂ Emissions (kg/kg)	Costs (USD/kg)
PC	0.8198 [10]	0.13500 [15]
DSP	0.0027 [10]	0.00618 [16]
SP	0.3800 [17]	0.92000 [18]

3. Results and Discussions

3.1. Compressive Strength

Figure 3 exhibits the compressive strength of cement pastes with different DSP replacement levels at 7 days, 28 days, and 60 days. It can be found that the 7-day compressive strength decreases as the DSP content increases, while the 28-day and 60-day compressive strength first increases and then decreases. The control group (CP-0DSP) has the highest compressive strength (43.3 MPa) at 7 days, and the 7-day compressive strength of the CP-20DSP, CP-40DSP, and CP-60DSP sample decreases by 9.47%, 32.25%, and 48.04%, respectively. The reason is that cement is replaced by DSP, and then the hydration product content decreases. Although the physical effects of DSP, such as the filling effect and nucleation effect, can compensate for part of the strength loss [19], the early positive effect of low-activity DSP is not obvious, leading to a decrease in compressive strength. Compared with the control group, the compressive strength in the CP-20DSP sample increases by 5.92% and 3.32% at 28 days and 60 days, respectively. DSP reacts with calcium hydroxide (CH) to generate additional C-S-H at later ages as a result of the pozzolanic effect of DSP [20]. Meanwhile, the nucleation effect of DSP also promotes cement hydration, enhancing its compressive strength. In addition, adding DSP can fill the voids between cement particles and increase the packing density of the specimen due to the small particle size of DSP [9]. However, the 28-day and 60-day compressive strength in the CP-60DSP sample display a decrease of 22.71% and 19.93%, respectively, compared with the CP-0DSP sample because the cement and the hydration product content are significantly reduced when a large amount of DSP is added. The reduction in compressive strength due to a lesser quantity of hydration products cannot be fully mitigated by the positive impact of DSP.

**Figure 3.** Influence of DSP on the compressive strength of cement paste.

3.2. Heat Evolution

Figure 4 depicts the DSP influence on the hydration heat of cement pastes. The second heat flow peak of the cement paste containing DSP is significantly shifted to the left, and the time from the beginning of the test to the appearance of the second heat flow peak is shortened. The large specific surface area of DSP can provide additional nucleation sites for cement hydration [21], thereby accelerating hydration. In addition, the cumulative heat release per gram of binder in the CP-20DSP sample is 98.34% of the CP-0DSP sample, showing that the addition of 20 wt.% DSP can make up for the hydration heat loss due to the reduction in cement content. However, after further increasing the DSP substitution

level, the cumulative heat release undergoes a notable reduction. Compared with the CP-0DSP sample, the cumulative heat release of the CP-40DSP and CP-60DSP samples exhibits a decrease of 19.93% and 47.75%, respectively. Even though DSP aids in hydration, a significant reduction in the hydration reaction degree of the sample occurs when cement is replaced with a high quantity of DSP, leading to a notable decline in cumulative heat release.

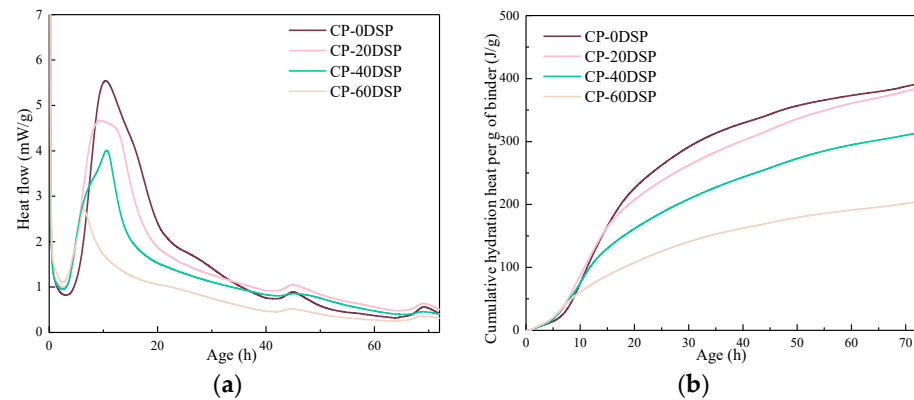


Figure 4. Influence of DSP on the hydration heat evolution of the pastes, (a) heat flow, and (b) cumulative hydration heat.

3.3. Pore Structure

Figure 5 displays the pore size distribution of the 28-day sample measured using MIP technology. The pore structures can be classified into harmless pores (<20 nm), less harmful pores (20–50 nm), harmful pores (50–200 nm), and more harmful pores (≥ 200 nm) [22,23]. The porosity of CP-0DSP, CP-20DSP, CP-40DSP, and CP-60DSP samples is 0.0896, 0.0737, 0.0999, and 0.1210 mL/g, respectively. The CP-20DSP sample exhibits the lowest porosity, marking a 17.75% decrease compared to the CP-0DSP sample. Meanwhile, the addition of 20 wt.% DSP effectively increases the proportion of harmless and less harmful pores and reduces the proportion of more harmful pores, showing that an appropriate amount of DSP instead of cement has a positive impact on the microstructure. The reason is that DSP can promote cement hydration and generate more hydration products [24]. In addition, DSP can fill the voids and pores inside the paste to improve the density [25]. However, incorporating 60 wt.% DSP significantly increases the porosity of the sample, which is 35.04% higher than the CP-0DSP sample, and the proportion of harmful and more harmful pores in the sample increases significantly. DSP increases the effective water–cement ratio and then the relative amount of mixing water increases, resulting in an increment in the pores formed [4]. Meanwhile, the reduction in hydration product content leads to a loose internal structure.

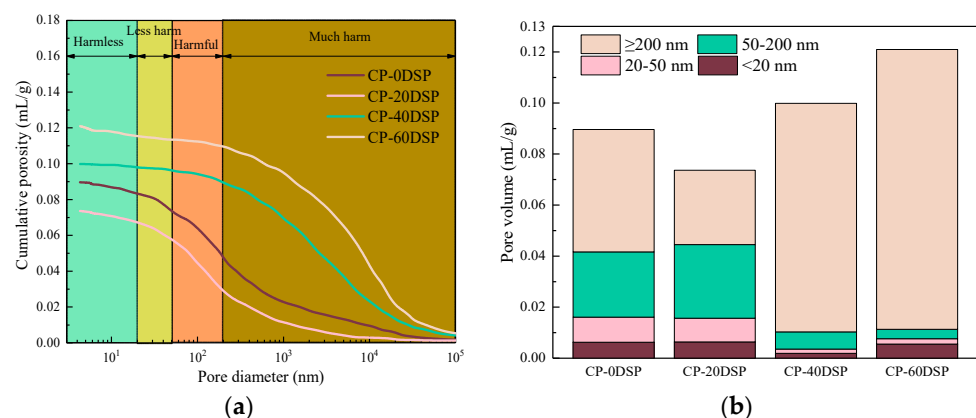


Figure 5. The pore structure of pastes at 28 days determined using MIP, (a) cumulative porosity, and (b) pore volume distribution.

3.4. Micromorphology

Figure 6 shows the SEM image of the 28-day sample. The CP-0DSP sample mainly contains fine needle-like AFt, flocculent or fluffy C-S-H, and CH crystals, as well as some pores. In comparison, the C-S-H is more continuous, and the connection between the matrices is tighter in the CP-20DSP sample. Meanwhile, the presence of a large amount of AFt makes the cement matrix more dense. DSP can provide additional nucleation sites for cement hydration and generate more hydration products. With the DSP content further increasing, the hydration product content decreases and a large number of DSP particles can be seen. In addition, DSP particles act as nucleation sites and are covered by deposited hydration products. There are obvious pores and voids in the matrix, and the matrix connection is relatively loose. When excess DSP replaces cement, fewer hydration products are generated, and the physico-chemical effects brought by DSP cannot fully compensate for the loss of strength caused by the reduction in cement.

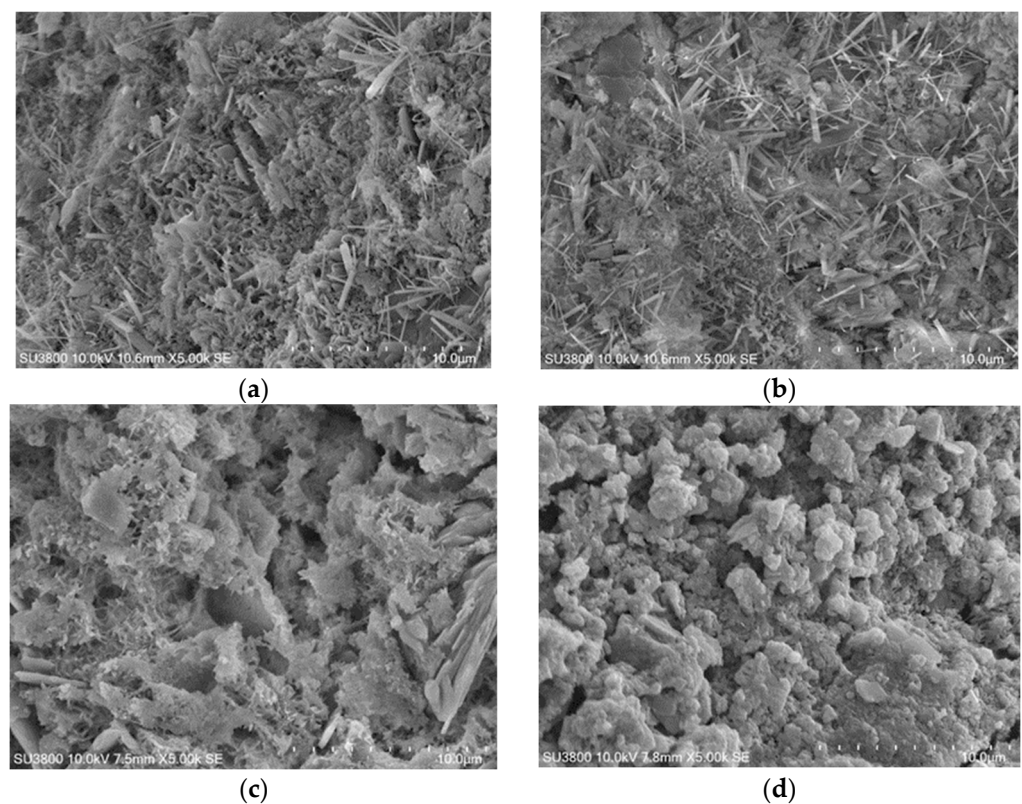


Figure 6. SEM images of cement paste with different DSP contents, (a) CP-0DSP, (b) CP-20DSP, (c) CP-40DSP, and (d) CP-60DSP.

3.5. Nanoindentation

Utilizing a lattice composed of a 10×10 grid arrangement (spacing $10 \mu\text{m}$), the nanomechanical properties and phase distribution of cement pastes are obtained using nanoindentation testing. According to the elastic modulus, different phases can be classified into pores ($\leq 8 \text{ GPa}$), low-density (LD) C-S-H ($8\text{--}20 \text{ GPa}$), high-density (HD) C-S-H ($20\text{--}34 \text{ GPa}$), CH ($34\text{--}50 \text{ GPa}$), and unhydrated particles (UP, $\geq 50 \text{ GPa}$) containing unhydrated cement and DSP [26]. Compared with the CP-0DSP sample, the elastic modulus distribution of the CP-20DSP sample undergoes a marked improvement, while the elastic moduli of CP-40DSP and CP-60DSP decrease, as shown in Figure 7.

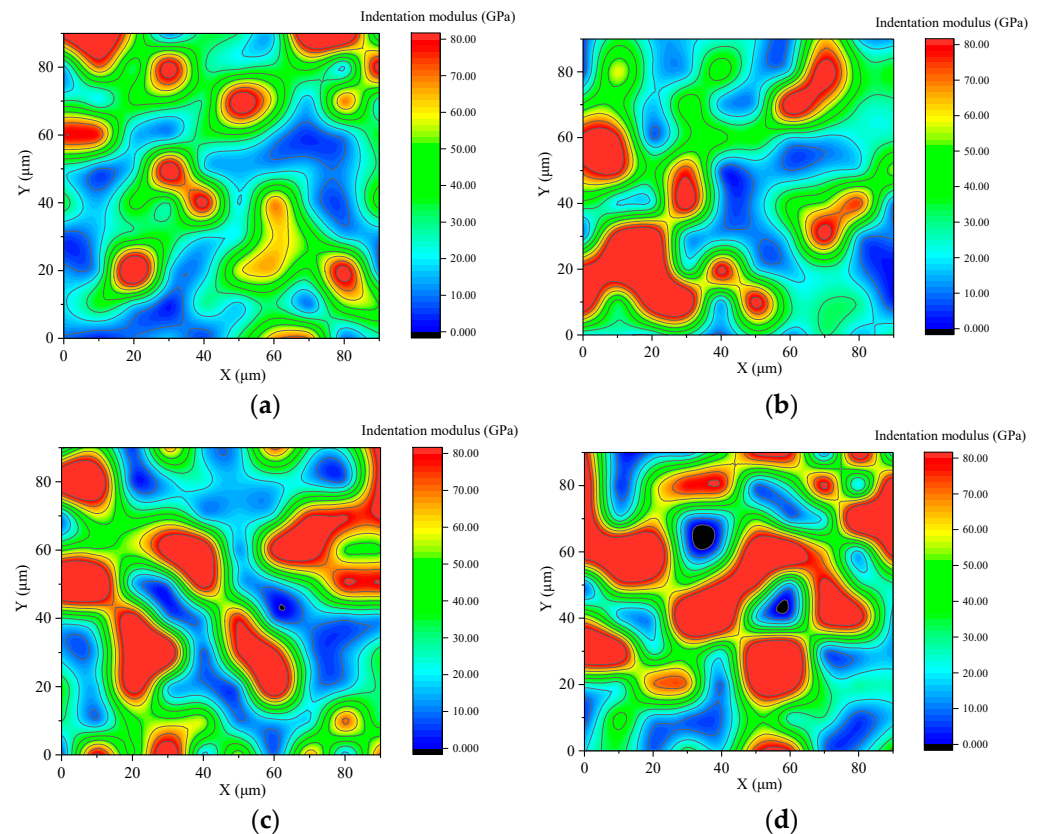


Figure 7. Indentation modulus distribution of the 28-day cement pastes, (a) CP-0DSP, (b) CP-20DSP, (c) CP-40DSP, and (d) CP-60DSP.

Gaussian function fitting is used to analyze the elastic modulus, as depicted in Figure 8. C-S-H exhibits the highest frequency, spanning from 33% to 52%, as it functions as the principal hydration product, responsible for imparting mechanical properties in cement pastes [26]. The C-S-H fitting peak in the CP-20DSP sample is observed to be higher than in the CP-0DSP sample, attributed to the pozzolanic effect that leads to the production of C-S-H. Concurrently, this results in an increased C-S-H density, further enhancing the mechanical properties of the pastes.

Figure 9 depicts the volume distribution of different phases of cement paste. Compared with the control group, the LD C-S-H and HD C-S-H volume fractions increase by 6.06% and 13.33%, respectively, in the CP-20DSP sample, suggesting that adding 20 wt.% DSP not only promotes the C-S-H formation, but also promotes the LD C-S-H transition to HD C-S-H. Meanwhile, the pore phase and CH phase volume fractions decrease by 23.08% and 14.29%, respectively. However, the C-S-H and CH volume fractions decrease significantly as the DSP substitution level further increases. The LD C-S-H and HD C-S-H volume fractions are reduced by 24.24% and 46.67%, respectively, in the CP-60DSP sample. Given that C-S-H is the primary contributor to compressive strength, especially HD C-S-H, a reduction in its content leads to a deterioration in the mechanical properties of the specimen. Additionally, compared with the CP-0DSP, the pore and UP phase volume fractions in the CP-60DSP sample increase by 30.77% and 68.00%, respectively, indicating that excessive DSP results in a relatively loose microstructure of the specimen, accompanied by a significant amount of UP phase.

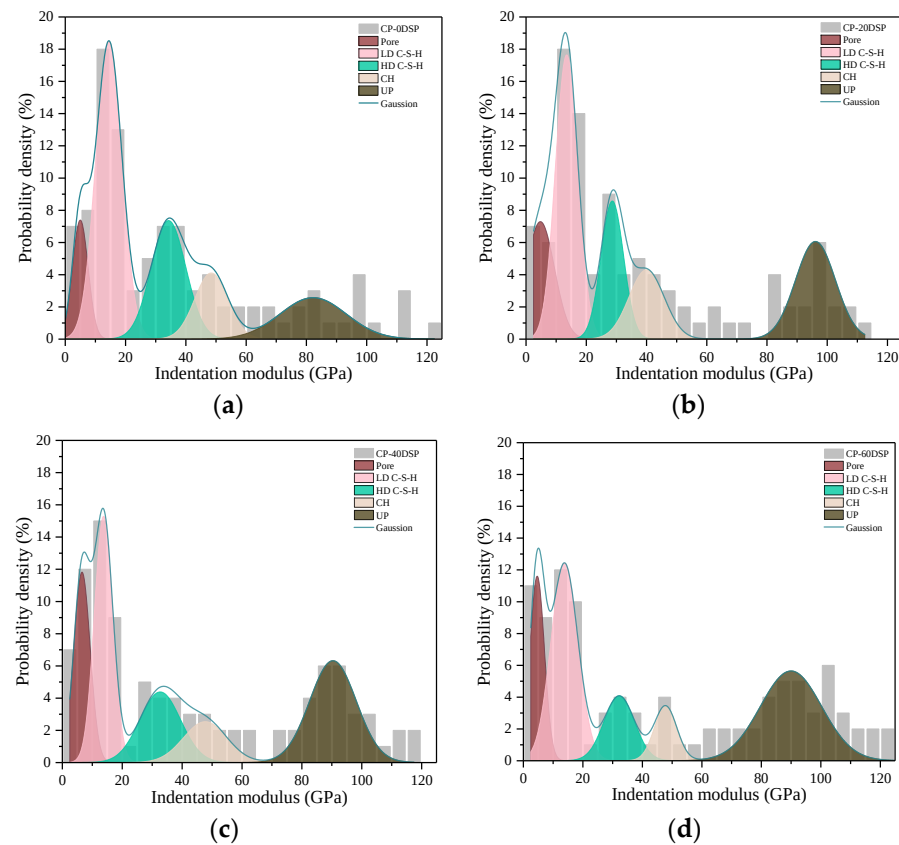


Figure 8. The frequency distribution of the elastic modulus of 28-day cement pastes, (a) CP-0DSP, (b) CP-20DSP, (c) CP-40DSP, and (d) CP-60DSP.

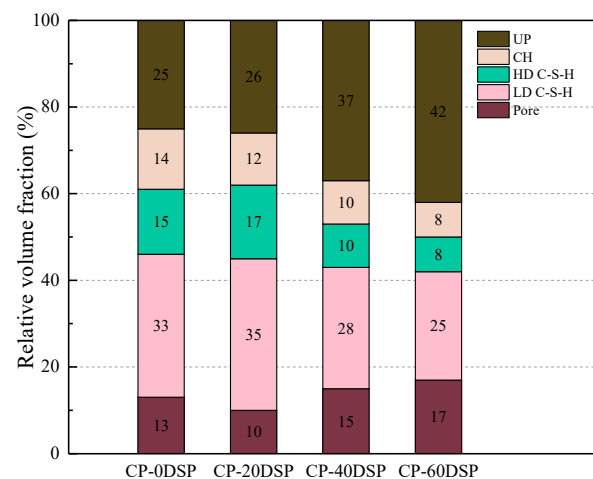


Figure 9. Volume fraction distribution of 28-day cement paste.

3.6. Environmental Assessment

Figure 10 shows that the CO₂ emissions and costs of cement pastes decrease inversely proportional to DSP content. Compared with the CP-0DSP sample, the CO₂ emissions of the CP-20DSP, CP-40DSP, and CP-60DSP samples are reduced by 19.93%, 39.86%, and 59.80%, respectively. This means that replacing cement with DSP can reduce the environmental burden of 1 m³ cement paste. Similarly, the costs of 1 m³ cement paste present a similar pattern of change as CO₂ emissions. Compared with the control group, the costs of CP-20DSP, CP-40DSP, and CP-60DSP samples are reduced by 19.05%, 38.11%, and 57.17%, respectively. Although incorporating 40 wt.%–60 wt.% DSP reduces the compressive strength, the CP-60DSP sample has the lowest CI coefficient and Cp coefficient, which are

13.02 kg/MPa·m³ and 2.29 USD/MPa·m³, respectively, considering the same compressive strength. The CI and Cp coefficients are reduced by 47.98% and 44.55%, respectively, compared with the CP-0DSP sample. The addition of 20 wt.%–60 wt.% DSP can develop an inexpensive and environmentally friendly cement paste, which is beneficial to alleviating the environmental burden and economic costs of the construction industry.

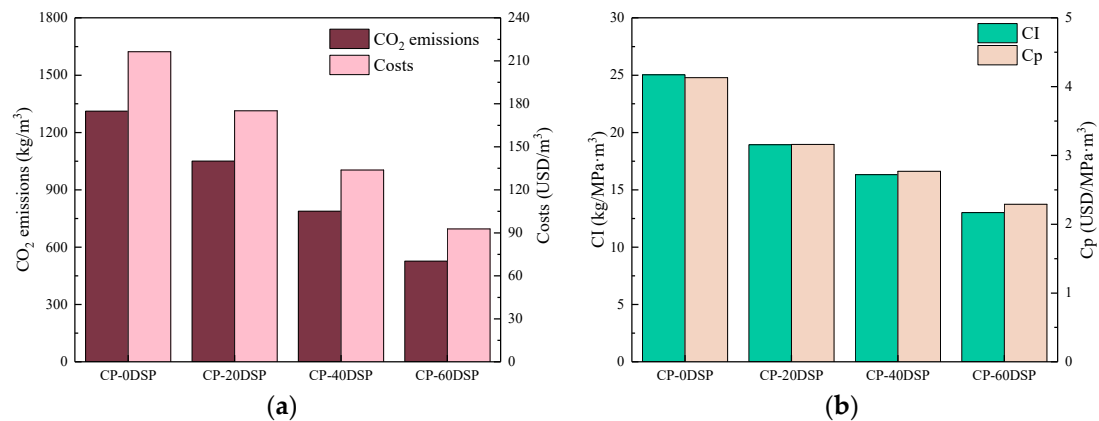


Figure 10. Effect of DSP on the economic cost and environmental effects of cement paste, (a) costs and CO₂ emissions, and (b) CI and Cp coefficients.

4. Conclusions

(1) Although incorporating DSP reduces the 7-day compressive strength of cement paste, the addition of 20% DSP can increase the 28-day and 60-day compressive strength of cement paste, which increases by 5.92% and 3.32%, respectively, compared with the control group.

(2) Adding 20 wt.% DSP can promote cement hydration. The time from the beginning of the test to the appearance of the second heat flow peak shortens as the DSP substitution level increases. Compared with the CP-0DSP sample, the cumulative heat release for the CP-40DSP and CP-60DSP samples is significantly lower, showing a decrease of 19.93% and 47.75%, respectively, except for the CP-20DSP sample.

(3) The addition of 20 wt.% DSP can effectively improve the pore structure of the sample. Compared with the control group, the porosity is reduced by 17.75%, while the proportion of harmless and less harmful pores is significantly increased, and the proportion of more harmful pores is reduced in the CP-20DSP sample.

(4) Adding 20 wt.% DSP can promote the conversion of CH to C-S-H, increase the C-S-H phase volume fraction, and reduce the pore phase volume fraction. However, excess DSP significantly increases the pore phase and UP phase volume fraction, while the C-S-H volume fraction decreases.

(5) Due to the low CO₂ emissions and low cost of DSP, the CO₂ emissions and costs are significantly reduced in the sample added with 20 wt.%–60 wt.% DSP. It is worth noting that CP-60DSP exhibits the lowest CI (13.02 kg/MPa·m³) and Cp (2.29 USD/MPa·m³) coefficients.

(6) Although this paper has conducted some work on applying DSP in cement-based materials, the effect of adding DSP on durability has not yet been clarified. At the same time, the composition of DSP also varies due to the different sources of DS, which needs further discussion.

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Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

Conflicts of Interest: Authors Jian Wang and Zhihui Yao were employed by the company Huahui Construction Group Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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