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Recycling Waste Molecular Sieves as Sustainable Additives in Concrete: A Technical-Environmental Assessment

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1. ABSTRACT

The management of industrial waste poses a significant environmental challenge in the modern era. This study explores the potential of recycling waste molecular sieves as a sustainable additive in concrete production. Using a comprehensive range of advanced analytical techniques, including XRF, XRD, FESEM, TEM, EDAX, BET-BJH, FTIR, TGA, and TPD, the physicochemical properties of this industrial waste were thoroughly characterized. The results revealed that gray and white waste molecular sieves primarily consist of aluminosilicate compounds and are free from hazardous environmental elements. Grinding up to 30,000 rpm significantly increased the amorphous phase, enhancing pozzolanic reactivity. The high specific surface area (140.9 m^2/g) and suitable porous structure indicate the high potential of this material for use in concrete. TPD analyses confirmed the presence of strong basic sites, ensuring compatibility with the alkaline environment of cement. These findings represent a significant step toward sustainable industrial waste management and the production of green construction materials.

Keywords: Waste Molecular Sieve, Industrial Recycling, Sustainable Concrete, Environmental Assessment, Pozzolan.

2. INTRODUCTION

The use of industrial waste in construction has gained attention as a sustainable and economic solution. Spent molecular sieves, despite losing crystallinity and accumulating carbon impurities during industrial use, retain high specific surface area and active basic sites [1, 2]. The spent molecular sieve used in this study is type 3A, originally used in natural gas dehydration. Its pore size is about 3 Å (<1 nm), classifying it as microporous [3].

3. MATERIALS AND METHODS

3.1. Materials

XRD (EQUINOX3000, Cu-Kα, 5-90°), FESEM (TESCAN Mira 3-XMU), EDAX (VEGA II), TEM (Philips EM208S), BET-BJH (ChemBET 3000, N_2), TPD-NH_3/CO_2 (Micrometrics 2910), FTIR, and TGA were used for characterization.

3.2. Material preparation

Grinding was performed using a planetary ball mill, and the total number of cycles (30,000 cycles) was recorded as the device's output criterion. All balls inside the device were used under standard conditions. Optimization of grinding parameters (ball diameter, ball-to-powder ratio, time, and speed) is planned for future research.

4. RESULTS AND DISCUSSION

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4.1. XRF

X-ray fluorescence (XRF) analysis was used to identify the elements present in the waste molecular sieve. The results of this analysis are presented in Table 1. The gray molecular sieve mainly consisted of aluminum and silicon, with its LOI value reported at approximately 15.92%, indicating the presence of possible carbon compounds. The white molecular sieve mainly consisted of Al_2O_3 with an LOI of approximately 16.7%. The mixture of these two samples had a composition similar to the gray sample. The sum of the reported percentages does not reach 100% due to the lack of measurement of light elements (oxygen, carbon, hydrogen) in XRF analysis. The remaining approximately 5% corresponds to bonded oxygen and carbon compounds, which have been confirmed by TGA and LOI analyses. (Table 1)

4.2. XRD

X-ray diffraction (XRD) analysis was performed to investigate the crystalline structure of the molecular sieves (Figure 1). After ball milling at 30,000 cycles and separation with various sieves, the crystalline and amorphous percentages were calculated using OriginPro software. The results showed that the highest amorphous content was related to the sample milled at 30,000 cycles and retained on sieve No. 100. Milling at 30,000 rpm led to a slight increase in the amorphous phase (from 3% to approximately 4.5-17.5% depending on particle size distribution), which can be chemically effective in pozzolanic reactivity, although numerically limited. Increasing the milling speed to 56,000 cycles did not create a significant change in the amorphous content; therefore, 30,000 cycles is suitable for economic and operational use.

Table 1. XRF of grined molecular sieves.

Elements	White	Gray	Mixture
L.O.I	16.7	15.92	15.64
Na_2O	0.253	5.872	6.047
MgO	0.136	2.45	2.545
Al_2O_3	81.822	24.27	24.67
SiO_2	0.571	39.134	38.39
P_2O_5	0.041	0.077	0.06
SO_3	0.072	0.189	0.2
Cl	0.025	0.041	0.101
K_2O	0.276	10.197	10.57
CaO	0.073	0.595	0.588
TiO_2	-	0.145	0.153
Fe_2O_3	0.018	0.016	1.036
Ni	0.005	0.005	-
Zn	0.004	0.004	-
Rb	-	0.003	-
Sr	-	0.004	-
Zr	-	0.004	-
Ga	0.001	-	-

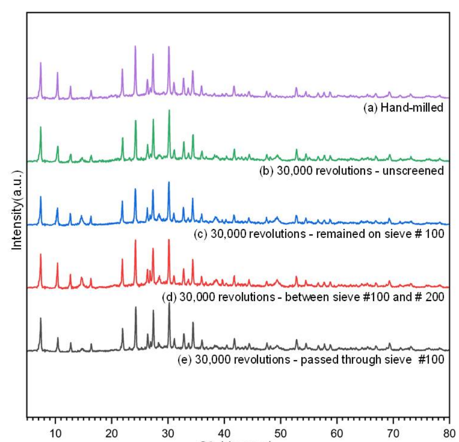

Figure 1. XRD grined molecular sieves.

4.3. FESEM and TEM

FESEM images showed that milling at 30,000 cycles increased particle agglomeration, indicating the transformation of part of the crystalline structure into an amorphous phase. The highest agglomeration was observed in the sample retained on sieve No. 100, which was consistent with XRD results. TEM images of the sample milled at 30,000 cycles and retained on sieve No. 100 revealed that the particles contain both crystalline and amorphous portions, with the amorphous phase appearing as agglomerated particles (Figure 2). This structure can enhance the reactivity of the material in concrete production.

4.4. Preliminary Investigation of Concrete Performance Containing Molecular Sieves

To preliminarily evaluate the applicability of waste molecular sieves in concrete, concrete specimens containing this material were prepared and tested in three different modes: aggregate replacement, powder as a substitute for sand, and cement replacement. The summarized results in Table 4 show that limited use of this material can improve concrete permeability and durability while maintaining acceptable strength.

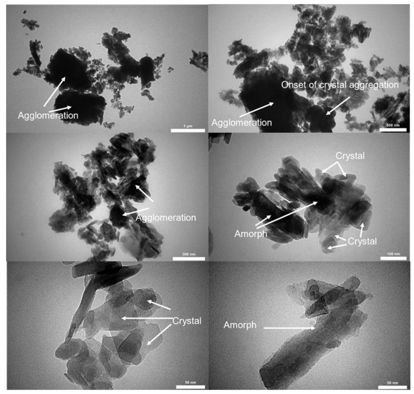

Figure 2. TEM of grined molecular sieve remained on sieve#100.

Table 2. Performance results of concrete containing waste molecular sieves

Application Mode	Parameter	Control Value	Optimum Value	Change	Allowable Range
Aggregate Replacement	Compressive strength (28 days, C390) - MPa	36.64	30	0.7% decrease per 1% replacement	15-20%
	Chloride permeability (C390) -C	67	224.7 (at 25%) 795.7 (at 100%)	3.5-fold increase at 100%	Up to 25%
Powder as Sand Replacement	Compressive strength (28 days, C390+50) - MPa	36.64	36	Neutral effect	Up to 150 g/m³
	Water penetration depth (C390) - mm	28	15 (at +50) / 0 (at +150)	46-100% reduction	25-50 kg/m³
	Chloride permeability (C390) - C	67	269.6 (at 25%) / 128.2 (at 50%)	52% reduction at 50%	Up to 50 kg/m³
Cement Replacement	Compressive strength (28 days, C390) - MPa	36.64	35	Significant decrease above 10%	Up to 5%
	Water penetration depth (C390) - mm	28	20 (at 5%) 40 (at 20%)	43% increase at 20%	Up to 5%
	Chloride permeability (C390) - C	67	93.9 (at 5%) 887 (at 30%)	13-fold increase up to 30%	Up to 5%

5. CONCLUSION

Milling at 30,000 rpm increased the amorphous phase (up to 17.5%), and the high surface area (140.9 m²/g), mesoporosity (88.5%), and strong basic sites confirm the potential of waste molecular sieves for concrete. Limited replacement (aggregate: 15-20%, sand: ≤150 kg/m³, cement: ≤5%) maintains acceptable properties while improving durability.

6. REFERENCES

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