

Recycling lithium mining waste into geopolymeric materials

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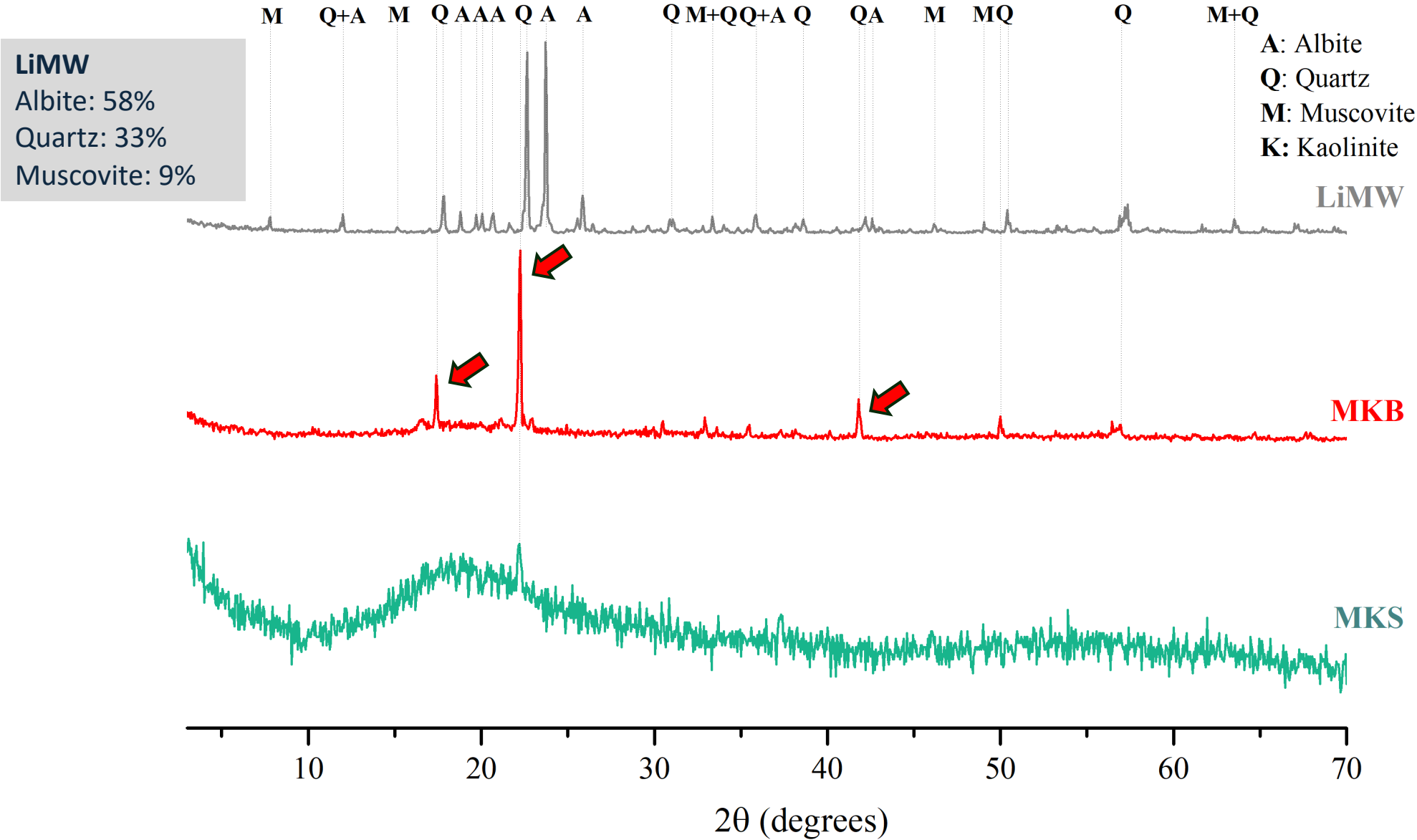
Geopolymer syntheses using lithium mining waste and two sources of metakaolin

Table 1. Chemical composition, particle size, and specific surface area of the metakaolins and the lithium mining waste.

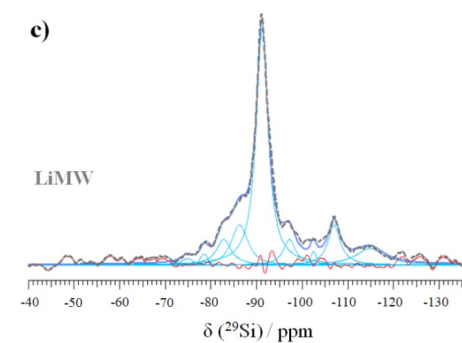
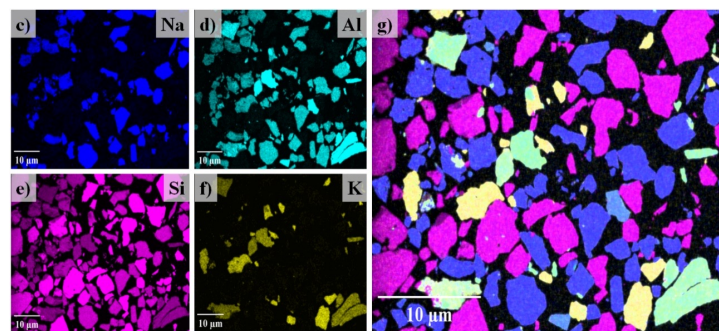
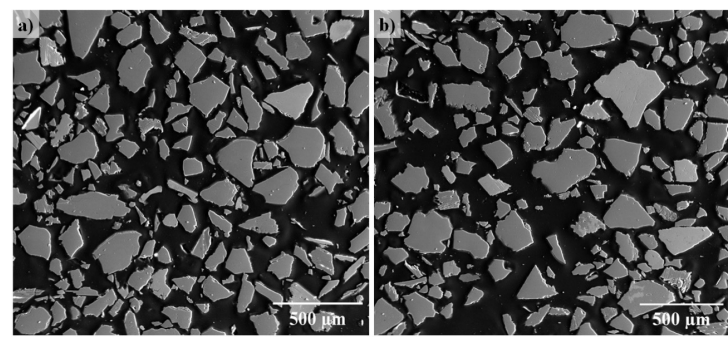
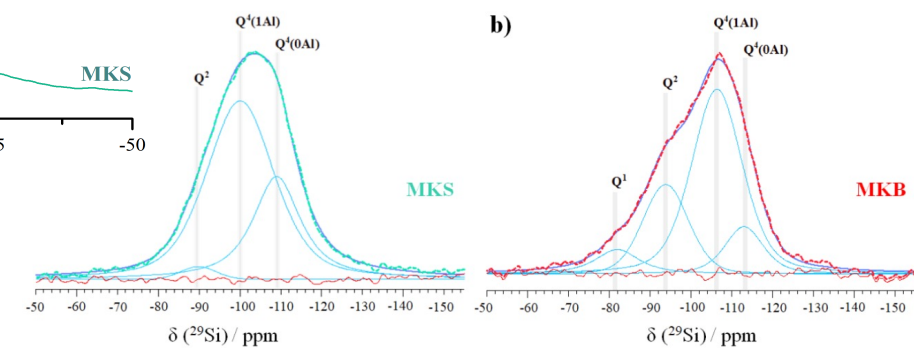
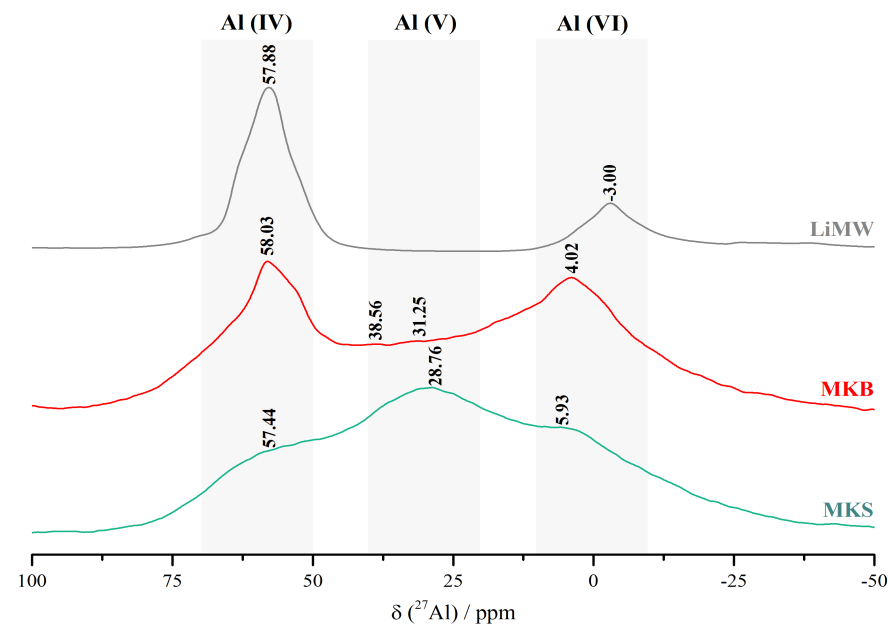
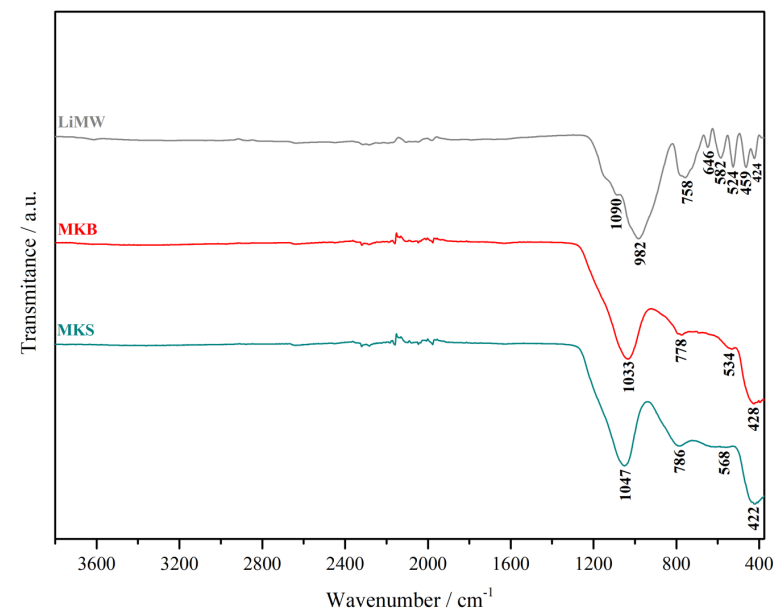
Sulfal metakaolin (MKS)				Metakaolin of Brazil (MKB)				Lithium mining waste (LiMW)			
Composition		Oxide	Wt. %	Composition		Oxide	Wt. %	Composition		Oxide	Wt. %
		SiO ₂	52.40			SiO ₂	56.90			SiO ₂	71.20
		Al ₂ O ₃	43.37			Al ₂ O ₃	34.10			Al ₂ O ₃	16.21
Molar ratio Si/Al			1.07	Molar ratio Si/Al			1.48	Molar ratio Si/Al			3.89
M. Particle size (μm)			19.56	M. Particle size (μm)			28.90	M. Particle size (μm)			141.98
Specific surface area			15.64 m ² /g	Specific surface area			19.19 m ² /g	Specific surface area			0.22 m ² /g
L.O.I. at 900°C			0.10%	L.O.I. at 900°C			4.30%	L.O.I. at 900°C			0.65 %

✓ The alkaline solution used had a $\text{SiO}_2\text{:Na}_2\text{O}$ molar ratio of 1.8.

Raw materials characterization: X-ray diffraction



Raw materials characterization: FT-IR, SEM/EDS, and ^{27}Al and ^{29}Si MAS NMR

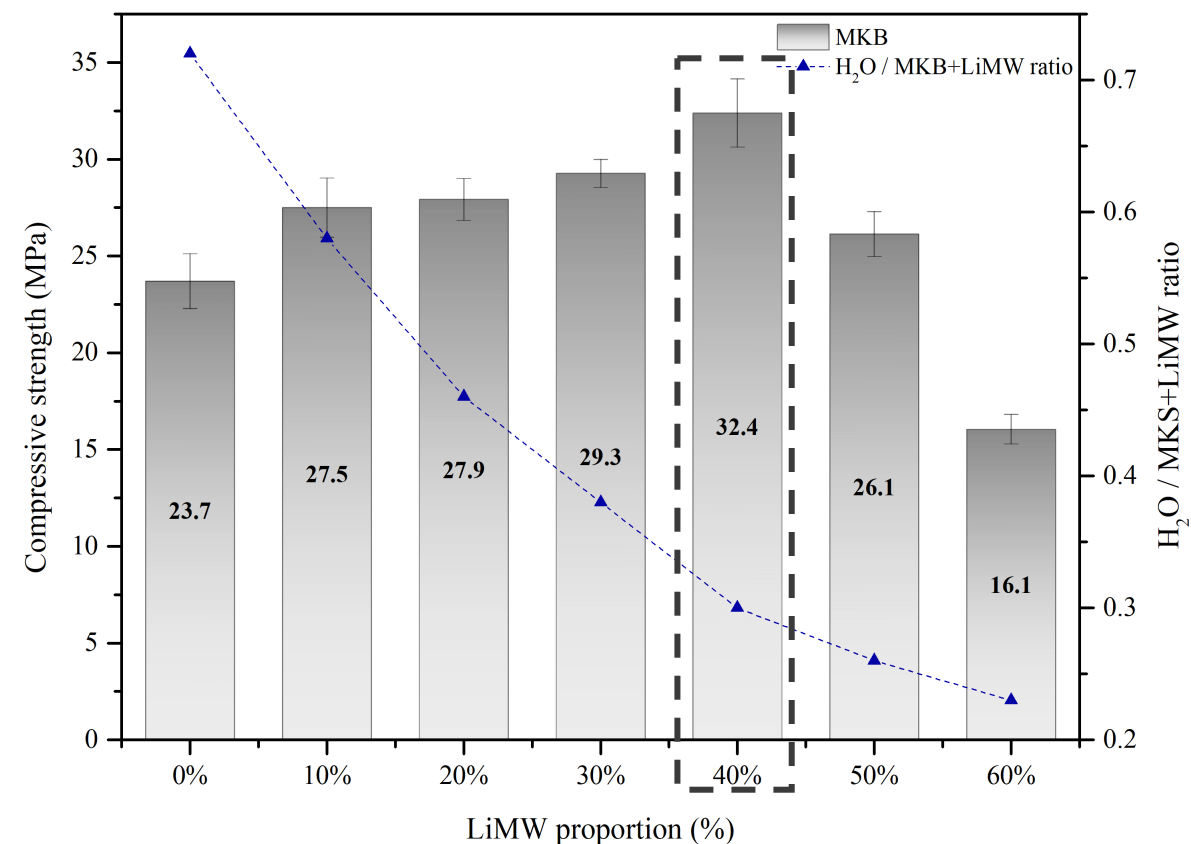
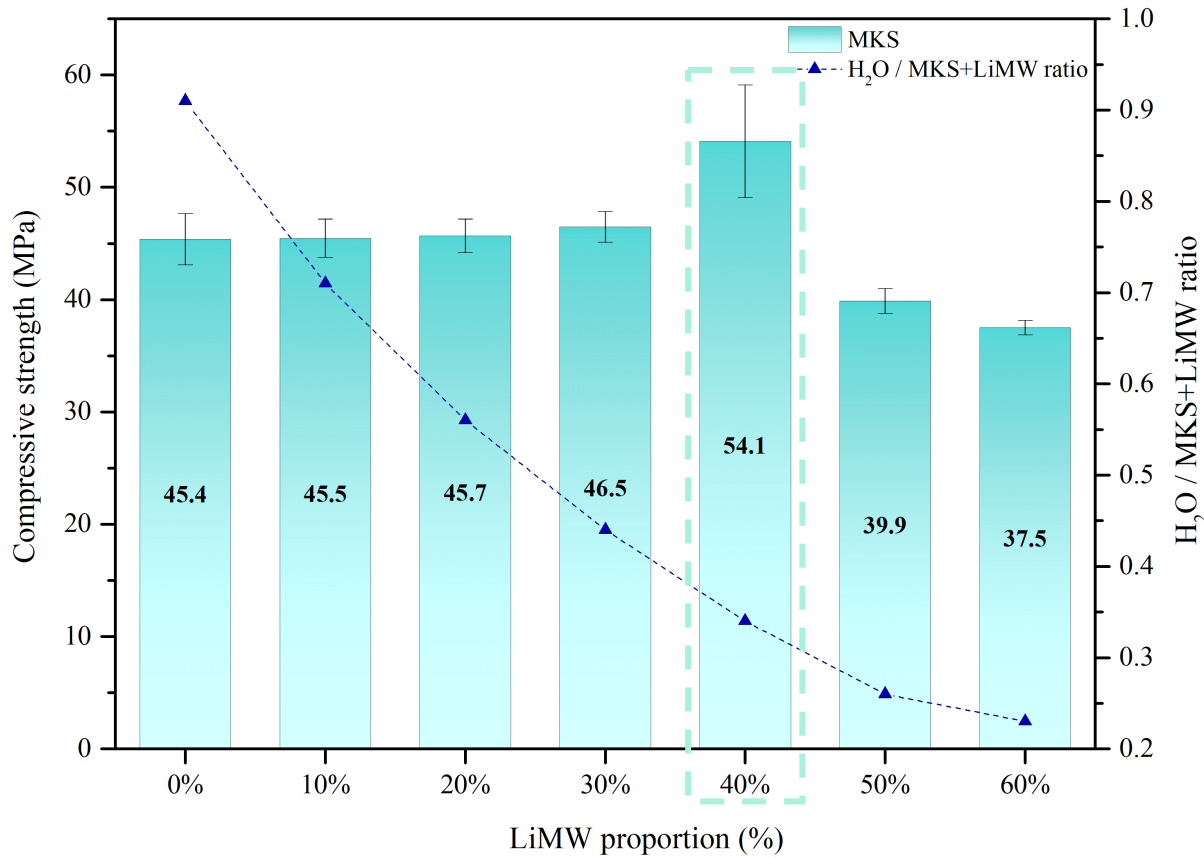


Metakaolin-based geopolymer formulations

Table 2. Geopolymer formulations with varying proportions of LiMW, MK, and AS (wt%)

Geopolymers	LiMW	MK	AS	Si/Al molar ratio	H ₂ O/MK+LiMW ratio
MKS.LiMW[0]	0	40.16	59.84	2.07	0.91
MKS.LiMW [10]	10	36.14	53.86	2.24	0.71
MKS.LiMW [20]	20	32.13	47.87	2.42	0.56
MKS.LiMW [30]	30	28.11	41.89	2.59	0.44
MKS.LiMW [40]	40	24.10	35.90	2.77	0.34
MKS.LiMW [50]	50	20.08	29.92	2.95	0.26
MKS.LiMW [60]	60	16.06	23.94	3.13	0.23
MKB.LiMW[0]	0	46.08	53.92	2.48	0.72
MKB.LiMW [10]	10	41.47	48.53	2.62	0.58
MKB.LiMW [20]	20	36.87	43.13	2.77	0.46
MKB.LiMW [30]	30	32.26	37.74	2.91	0.38
MKB.LiMW [40]	40	27.65	32.35	3.05	0.30
MKB.LiMW [50]	50	23.04	26.96	3.20	0.26
MKB.LiMW [60]	60	18.43	21.57	3.34	0.23

Influence of LiMW on geopolymers' mechanical properties



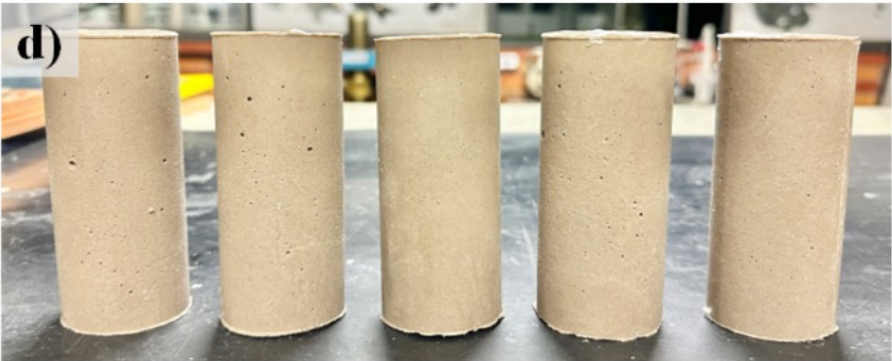
- ✓ The maximum compressive strength values were achieved utilizing MKS as a metakaolin precursor;
- ✓ Increasing the LiMW proportion in MK-based geopolymers, the molar ratio of Si/Al is increased by the replacement of MK with LiMW;
- ✓ The incorporation of LiMW, up to an adequate proportion, significantly enhanced the compressive strength in both syntheses.

Influence of LiMW on geopolymers' mechanical properties

MKS

MKB

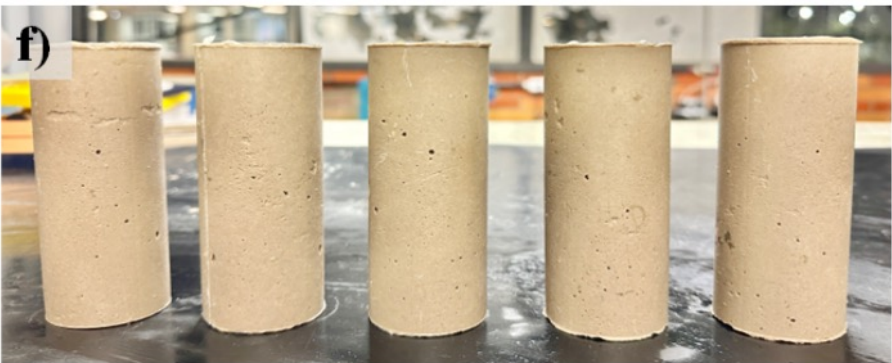
0 wt% of
LiMW



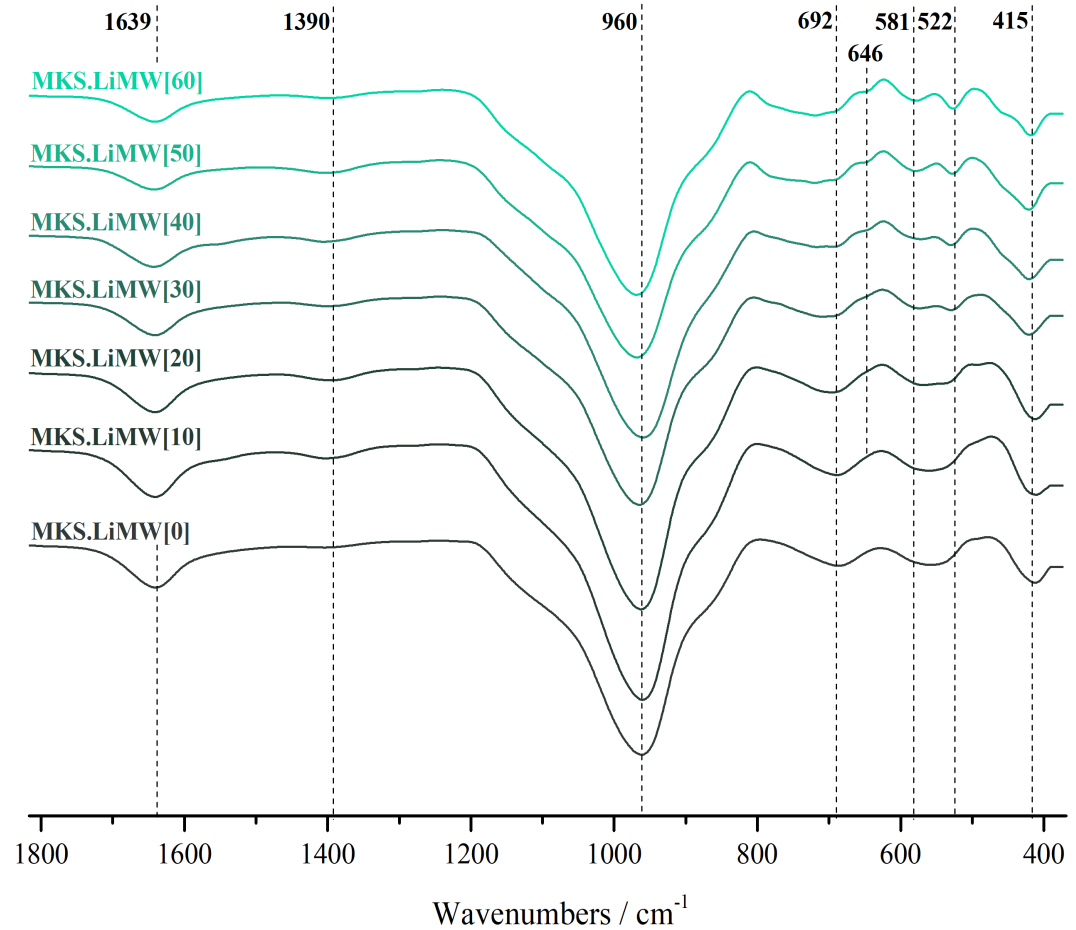
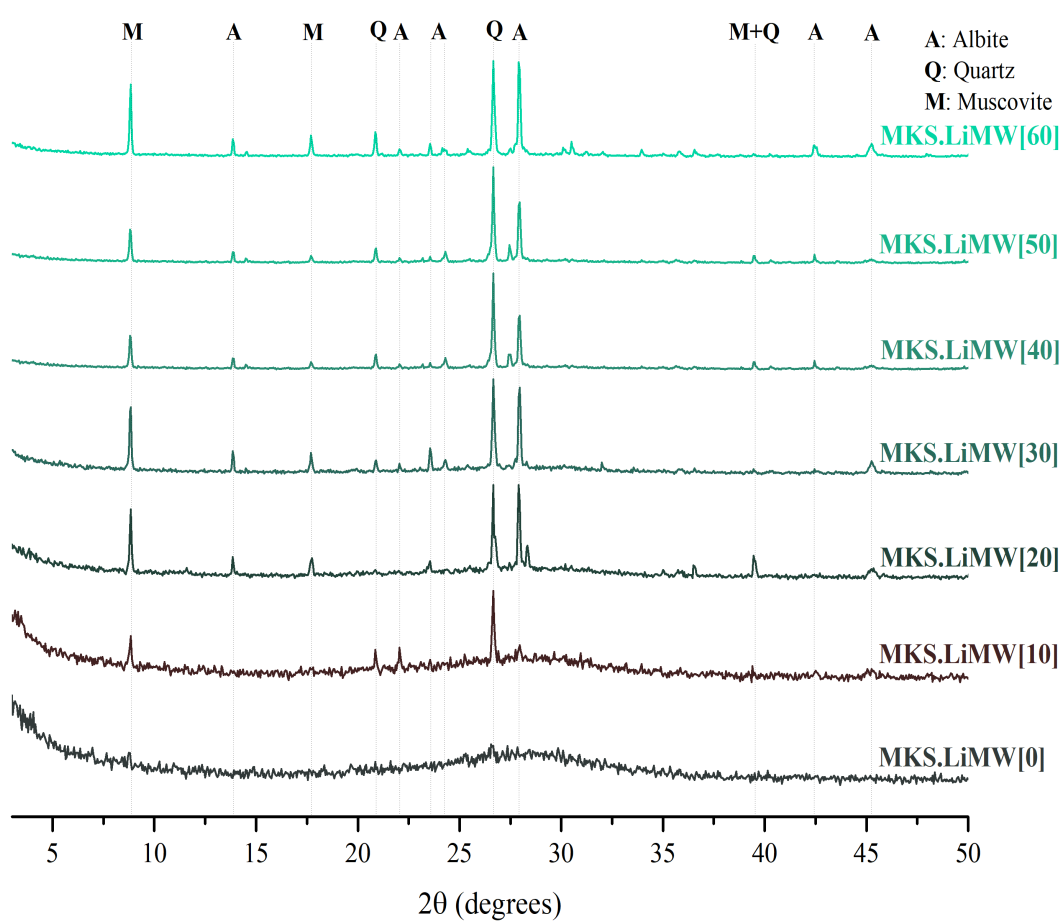
40 wt% of
LiMW



60 wt% of
LiMW

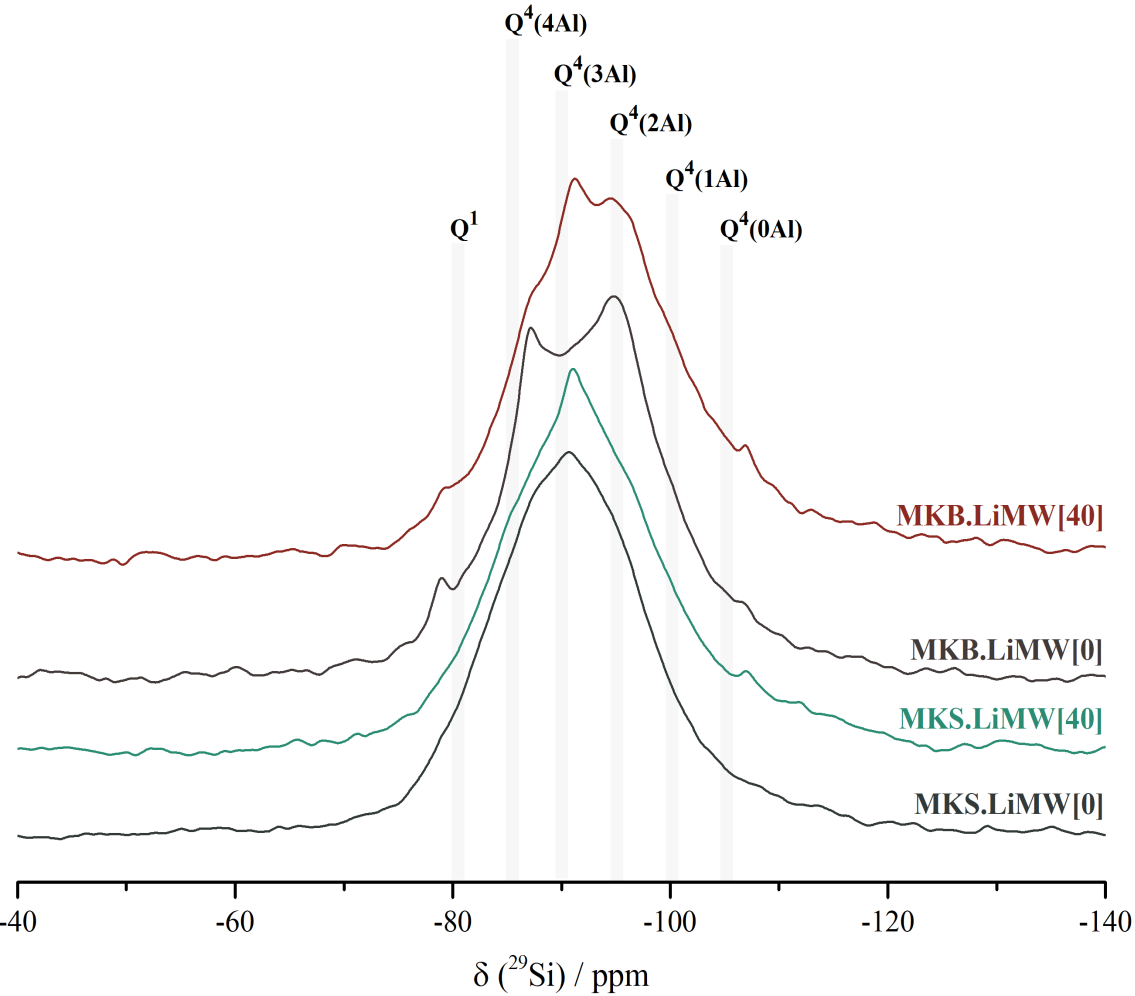
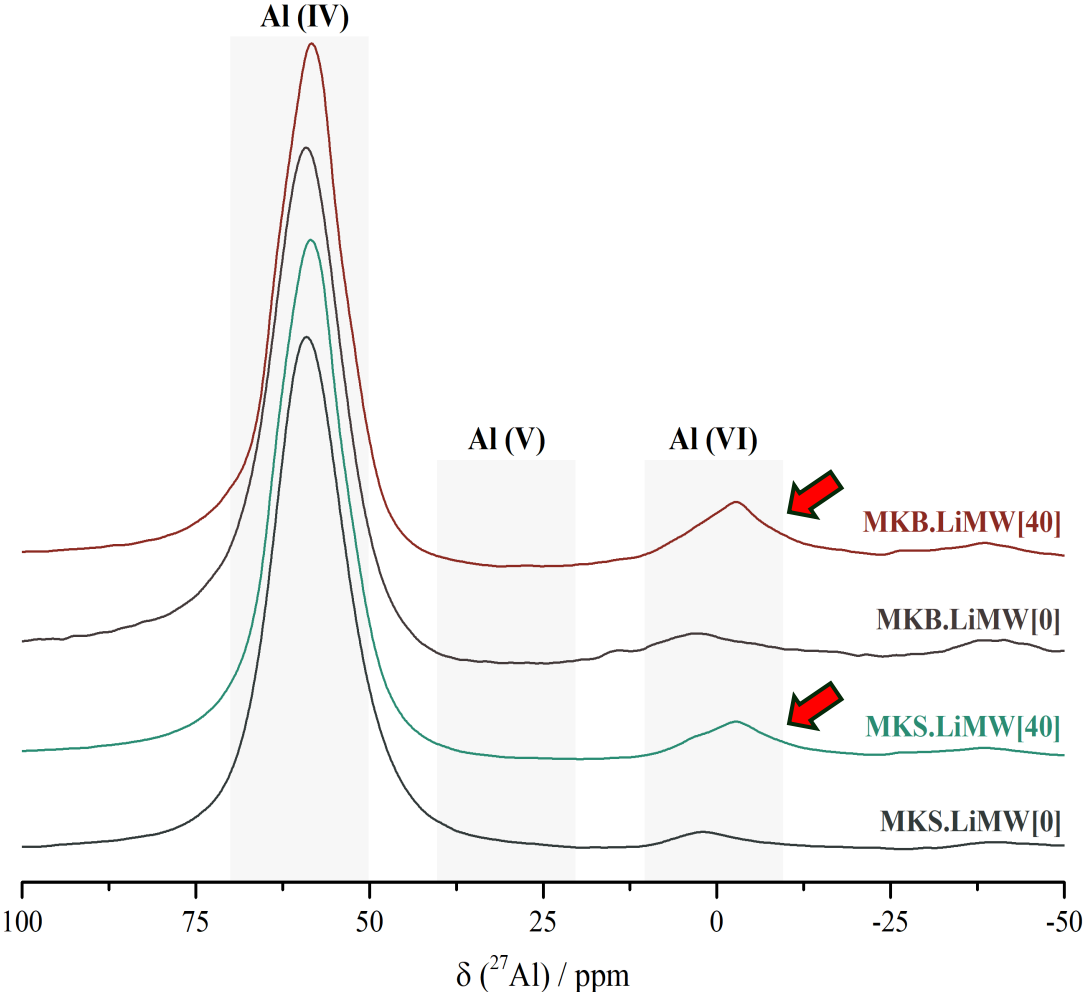


Geopolymer characterization: XRD and FT-IR



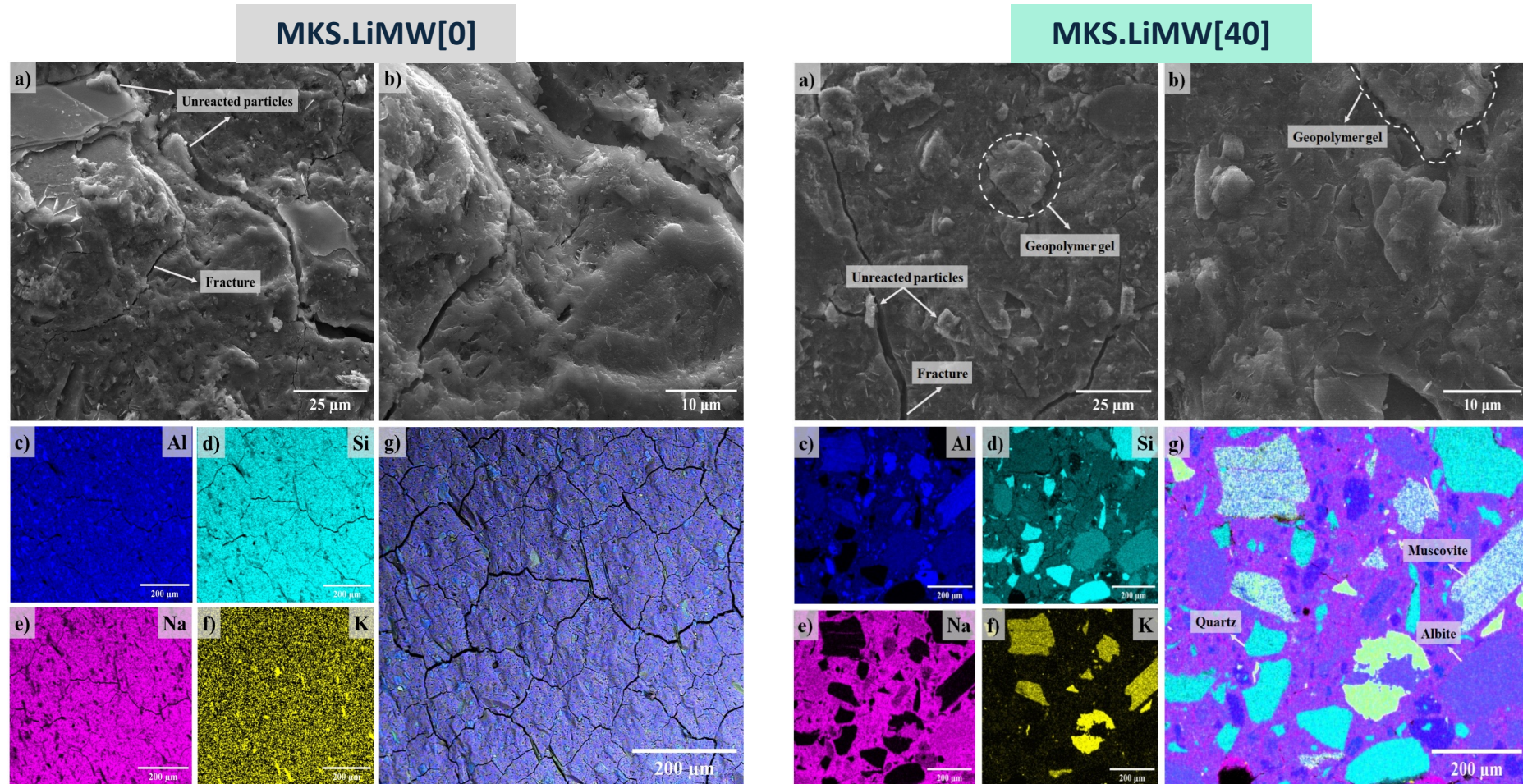
- ✓ XRD: As the LiMW proportion increases, the intensity of well-defined diffraction peaks associated with albite, quartz, and muscovite also increases;
- ✓ FT-IR: The most intense band in the region of 960 cm⁻¹ corresponds to internal vibrations of Si-O-Si (Al) of the geopolymeric materials.

Geopolymer characterization: ^{27}Al and ^{29}Si MAS NMR spectroscopy



- ✓ ^{27}Al MAS NMR: The predominant resonance signal at approximately 60 ppm corresponds to the tetrahedrally coordinated aluminum [Al(IV)];
- ✓ ^{29}Si MAS NMR: Different molecular arrangements and degree of condensation of the tetrahedral silicon units.

Geopolymer characterization: SEM images and EDS analysis



- ✓ MKS.LiMW[0]: Dense morphology with a homogeneous distribution of Al, Si, Na and K on the surface;
- ✓ MKS.LiMW[40]: Dense microstructure and morphology – The unreacted LiMW particles contribute to the arrangement and filling of voids between the geopolymer micelles (Packing/Filler effect). EDS mapping clearly reveals the distinction between areas predominantly composed of geopolymer gel and the unreacted particles.



Conclusions

- ✓ In-depth investigation of the influence of the metakaolin aluminosilicate source, the incorporation of lithium mining waste as an aggregate, and the amount of the alkaline solution used in the geopolymer mixture on compressive strength and physico-chemical properties;
- ✓ These results highlight the applicability of LiMW as a viable component in geopolymeric binders, especially in materials applied to the civil construction sector;
- ✓ This work reinforces the relevance of proactive research aligned with the SDGs, focusing on the reducing carbon footprint, promoting sustainable construction practices, minimizing waste through recycling and reuse, and improving the environmental management of mining tailings disposal.

Scaling up geopolymer synthesis for application in civil construction materials

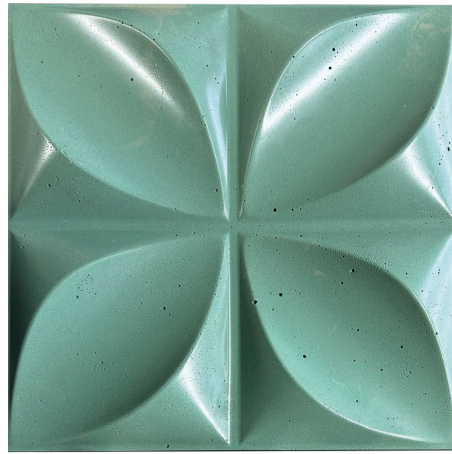


100kg/batch



300-500kg/batch

Products incorporating 40 – 60 wt% of lithium mining waste





Challenges in scaling up the geopolymers production

- ✓ Process adaptation – translating lab-scale synthesis procedures to pilot-scale equipment and batching systems;
- ✓ Controlling compressive strength to ensure mechanical performance consistency in scaled-up production;
- ✓ Mitigating surface cracking and efflorescence;
- ✓ Optimizing mixture design;
- ✓ Managing workability and setting time of the geopolymeric paste.

Acknowledgments & Contacts



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