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INFLUENCE OF REDISPERSIBLE POLYMER POWDER ON THE MICROSTRUCTURE AND PHASE COMPOSITION OF GYPSUM CONCRETE CONTAINING LIME WASTE

X.A. Mamatov, J.N. Akbarov
Fergana State Technical University

Abstract. The study investigates the structural and physicochemical characteristics of gypsum concrete containing lime waste and Redispersible Polymer Powder (RPP). The composition selected for the investigations consisted of 75% G-6 gypsum, 25% lime waste and 0.10% RPP relative to the mass of dry components. The physicochemical characteristics of the modified gypsum concrete were studied using thermogravimetric and differential thermal analysis, X-ray diffraction, infrared spectroscopy and scanning electron microscopy. Thermal analysis of gypsum concrete containing 0.10% RPP showed that the principal mass loss occurred in the temperature range of 301.08–601.24 °C, while a pronounced thermal effect was recorded at approximately 414.49 °C. X-ray diffraction analysis identified gypsum ($CaSO_4 \cdot 2H_2O$) as the main crystalline phase and quartz (SiO_2) as a secondary phase. The crystallite size of gypsum in the (020) direction was calculated to be approximately 100 nm. Comparative scanning electron microscopy of samples with and without RPP showed denser intercrystalline contacts and a decrease in structural voids in the modified material. Infrared spectra of the samples also demonstrated differences in characteristic absorption regions, indicating changes in the physicochemical structure of the gypsum matrix after the addition of RPP.

Keywords: gypsum concrete, lime waste, Redispersible Polymer Powder, RPP, microstructure, X-ray diffraction, thermal analysis, infrared spectroscopy.

1. Introduction

Gypsum-based materials are characterized by rapid setting and hardening and can be used for the production of construction products. At the same time, improving their structure and physicomechanical characteristics requires modification of the gypsum matrix and the use of mineral and polymer components.

In the investigated composition, lime waste generated during technical water treatment was used together with G-6 gypsum and Redispersible Polymer Powder. Experimental studies established a composition containing 75% G-6 gypsum, 25% lime waste and 0.10% RPP relative to the mass of dry components. For this composition, the flexural strength was 4.67 MPa and the compressive strength was 7.41 MPa.

The physicochemical study of the developed gypsum concrete was carried out to evaluate its phase composition, microstructure, hydration and crystallization processes. Thermogravimetric and differential thermal analysis, X-ray phase analysis, infrared spectroscopy, scanning electron microscopy and elemental analysis were used for this purpose.

The aim of this study is to compare the structural features of gypsum concrete with and without RPP and to evaluate the influence of the polymer additive on the phase and microstructural formation of the material.

2. Materials and Methods

The investigated gypsum concrete was prepared using G-6 grade gypsum, lime waste and Redispersible Polymer Powder. The amount of RPP accepted as optimal was 0.10% of the mass of dry components, corresponding to 1.0 kg of RPP per 1000 kg of dry components.

The composition used in further studies consisted of 75% G-6 gypsum, 25% lime waste and 0.10% RPP relative to the dry component mass.

The physicochemical characteristics of the samples were studied using thermogravimetric and differential thermal analysis, X-ray diffraction, scanning electron microscopy and infrared spectroscopy.

For thermal analysis, the samples were investigated by thermogravimetric and differential thermal methods. The temperature was increased at a rate of 10 °C/min during the thermogravimetric investigation.

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Infrared spectra were recorded in the 500–4000 cm^{-1} range in transmittance mode. The measurements consisted of 10 scans, and the spectral resolution was 4 cm^{-1}.

3. Results and Discussion

3.1. Thermal analysis

The thermal characteristics of gypsum concrete containing 0.10% RPP were studied using thermogravimetric and differential thermal analysis. For comparison, gypsum concrete without the polymer additive was also investigated.

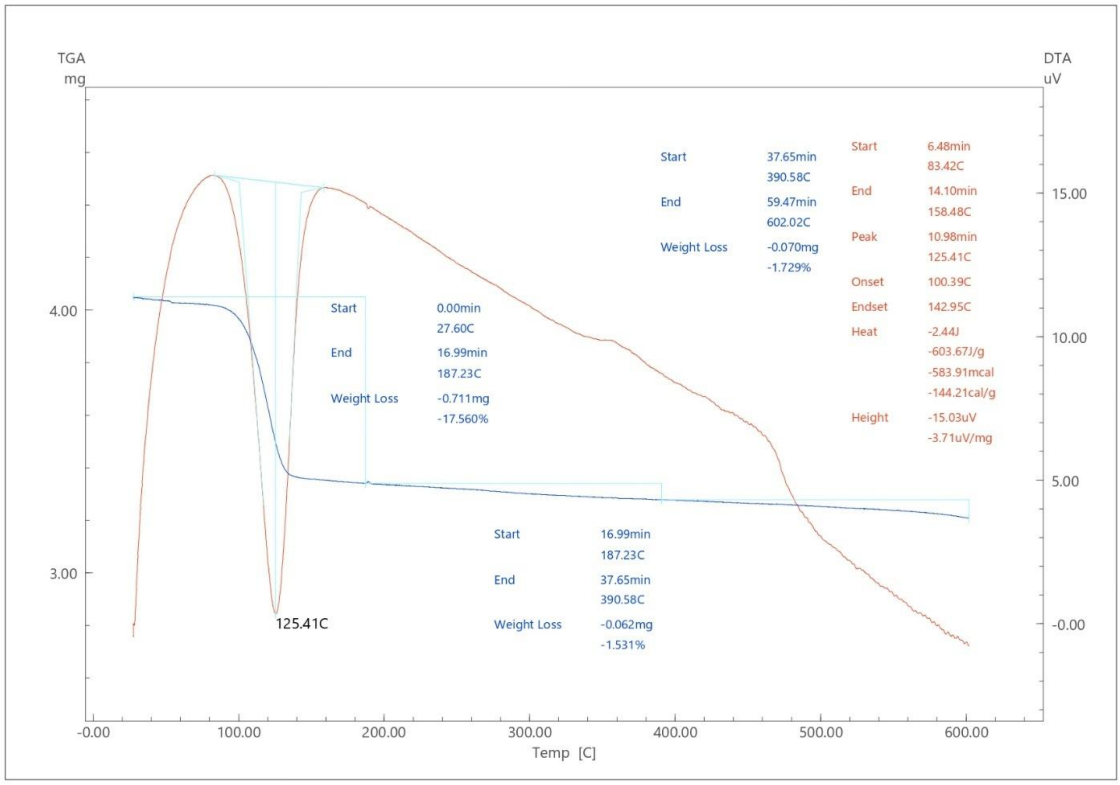


Figure 1. Thermogravimetric analysis of gypsum concrete without Redispersible Polymer Powder (RPP).

Table 1. Analysis of DTGA and DTA curve results for gypsum concrete without RPP.

No.	Temperature, °C	Mass loss, %	Decomposition rate, mg/min	Energy, µV·s/mg
1	50	0.540	5.41	7.22
2	100	1.008	12.65	7.18
3	134	1.007	19.10	6.14
4	197	0.125	17.25	5.87
5	200	14.898	14.76	6.68
6	300	1.987	18.85	6.14
7	400	1.689	13.86	6.11
8	500	1.618	8.56	6.08
9	585	1.763	1.09	5.77
10	600	1.763	2.49	6.03
11	700	2.95	2.35	5.92

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12	800	2.95	3.38	5.83
13	901	3.080	5.33	5.81

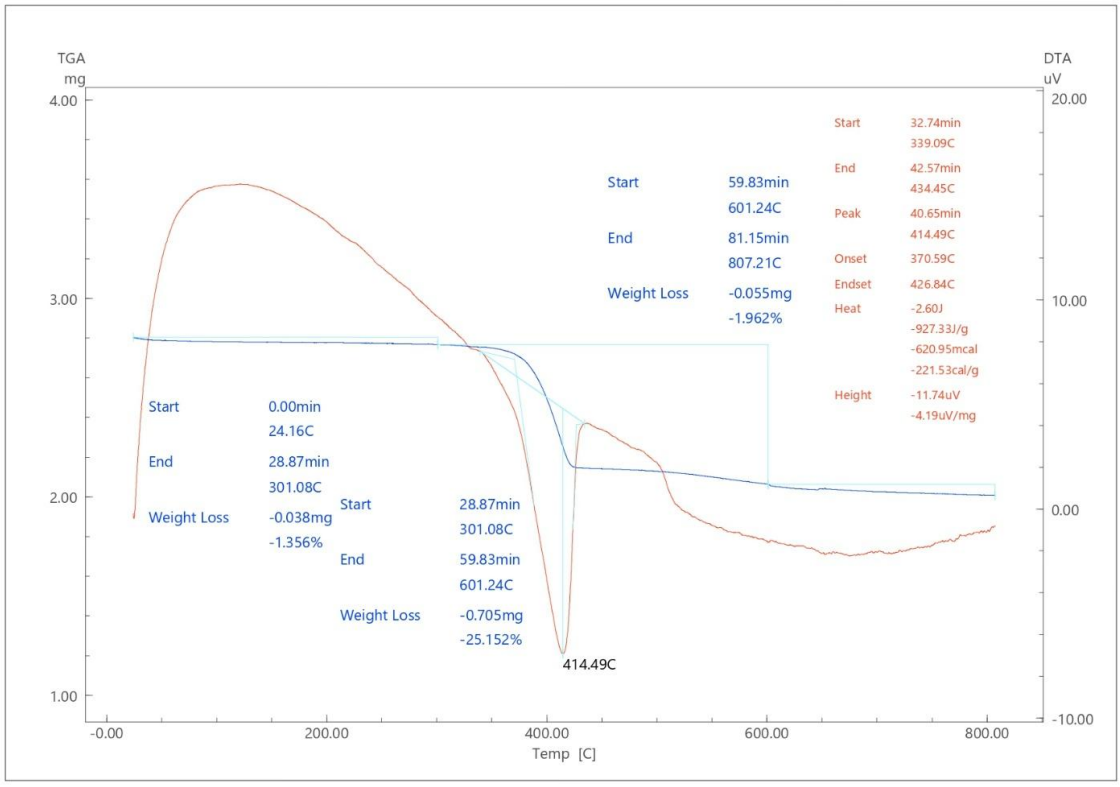


Figure 2. Thermogravimetric analysis of gypsum concrete containing Redispersible Polymer Powder (RPP).

In the RPP-modified gypsum concrete, three thermal decomposition stages were identified. The values shown in Tables 2–4 are reproduced from the experimental thermal analysis data.

Table 2. Stage I — loss of moisture and adsorbed gases.

Parameter	**Value**
Temperature range	24.16°C → 301.08°C
Time	0.00 → 28.87 min
Mass loss	−0.038 mg (−1.356%)

Table 3. Stage II — main thermal decomposition (dominant stage).

Parameter	**Value**
Temperature range	301.08°C → 601.24°C
Time	28.87 → 59.83 min
Mass loss	−0.705 mg (−25.152%)

Table 4. Stage III — high-temperature residual decomposition.

Parameter	**Value**
Temperature range	601.24°C → 807.21°C
Time	59.83 → 81.15 min
Mass loss	−0.055 mg (−1.962%)

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Table 5. Characteristics of the DTA peak recorded for the RPP-modified sample.

Parameter	Value	Description
Peak start	339.09°C / 32.74 min	Reaction starts
Peak end	434.45°C / 42.57 min	Reaction ends
Peak maximum	414.49°C / 40.65 min	Strongest reaction point
Onset temperature	370.59°C	Kinetic onset temperature
Endset temperature	426.84°C	Reaction end temperature
Heat effect	−2.60 J / −927.33 J/g	Strong exothermic
Peak height	−11.74 µV (−4.19 µV/mg)	Signal intensity

The principal mass loss in the RPP-modified gypsum concrete occurred in the temperature range of 301.08–601.24 °C. A pronounced thermal effect was recorded at approximately 414.49 °C.

3.2. X-ray diffraction analysis

X-ray diffraction analysis was used to determine the phase composition of the modified gypsum concrete. The main crystalline phase identified in the sample was gypsum ($CaSO_4 \cdot 2H_2O$), while quartz (SiO_2) was identified as a secondary phase.

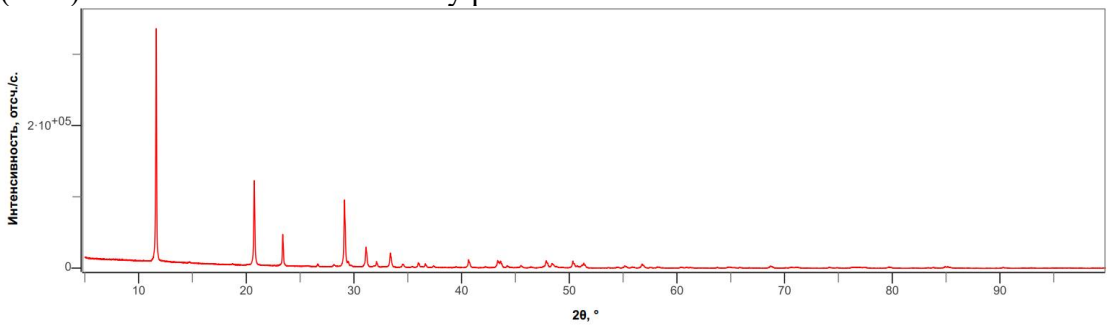


Figure 3. X-ray diffractogram of the investigated gypsum concrete sample.

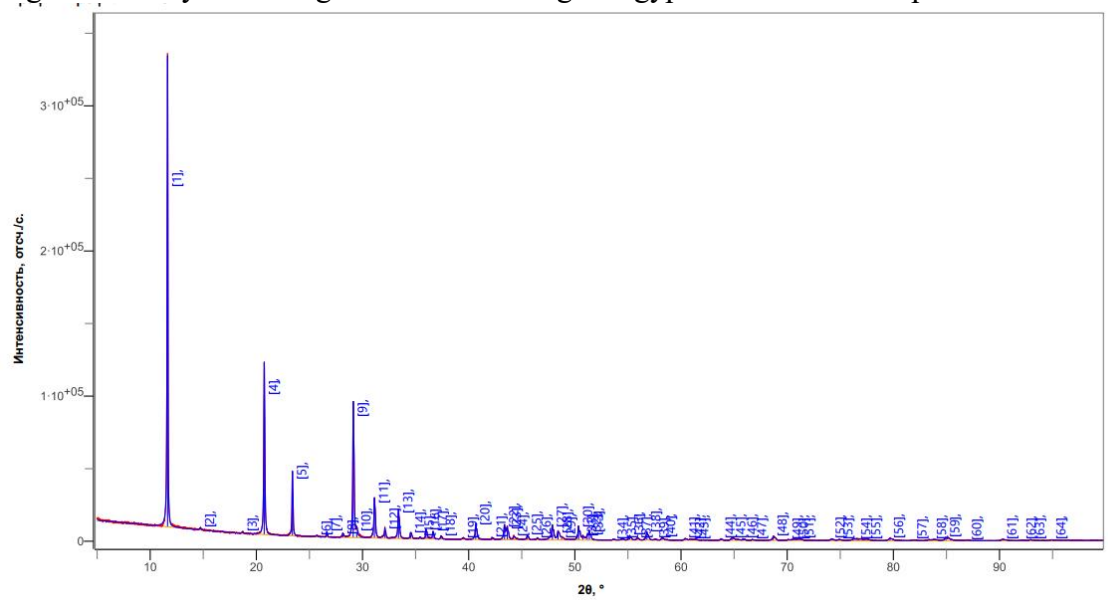


Figure 4. X-ray diffractogram of the investigated gypsum concrete sample with identified diffraction peaks.

Table 6. Main diffraction peaks of the gypsum phase.

Peak	2θ experimental	2θ reference	d (Å)	Hkl	Relative intensity

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1	11.63°	11.69°	7.600	(020)	100.00%
4	20.73°	20.74°	4.282	($1\bar{2}1$)	46.02%
5	23.38°	23.42°	3.802	(040)	16.04%
9	29.10°	29.15°	3.066	($1\bar{2}1$)/($2\bar{0}1$)	42.27%
11	31.10°	31.22°	2.873	(141)	14.63%
13	33.7°	33.41°	2.683	($2\bar{2}1$)	11.11%

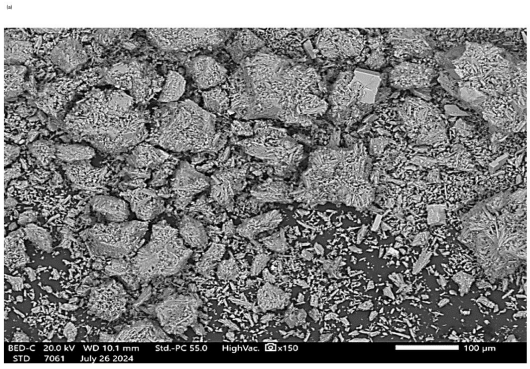
Table 7. Main diffraction peaks of the quartz phase.

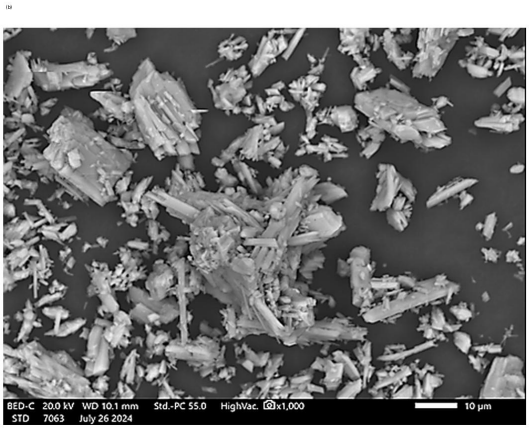
Peak	2θ experimental	d (Å)	Hkl	Relative intensity
7	26.61°	3.347	(101)	1.16%
17	36.62°	2.452	(110)	2.59%
19	39.48°	2.281	(200)	0.72%
30	50.32°	1.812	(112)	6.58%

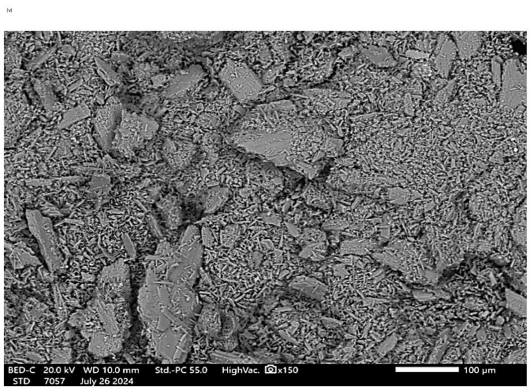
The crystallite size of gypsum in the (020) crystallographic direction was calculated to be approximately 100 nm. The diffraction data therefore identify gypsum as the principal crystalline phase and quartz as a secondary phase in the investigated sample.

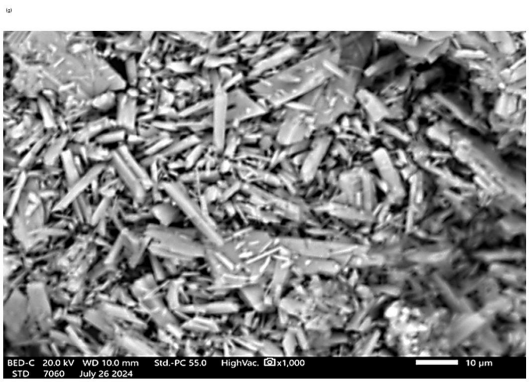
3.3. Microstructural characteristics

Scanning electron microscopy was used to compare gypsum concrete samples prepared with and without RPP. In the sample without RPP, gypsum crystals were predominantly larger and more dispersed. In the RPP-modified material, denser intercrystalline contacts and a reduction in structural voids were observed.









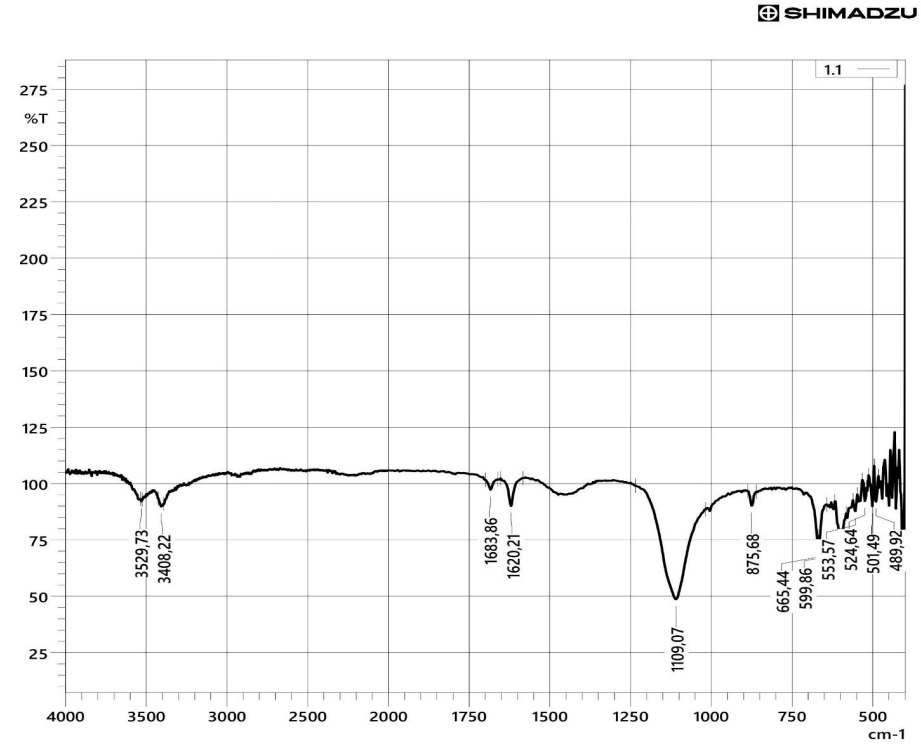
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Figure 5. SEM microstructure: (a) and (b) gypsum concrete without RPP; (v) and (g) gypsum concrete prepared with 0.10% RPP.

The electron-microscopic observations show that the RPP-modified sample has a denser arrangement of crystals than the unmodified sample. These observations were used to evaluate the influence of RPP on microstructural formation.

3.4. Infrared spectroscopic analysis

Infrared spectroscopy was used to compare gypsum concrete containing lime waste with and without RPP. The measurements were performed in the 500–4000 cm^{-1} range.



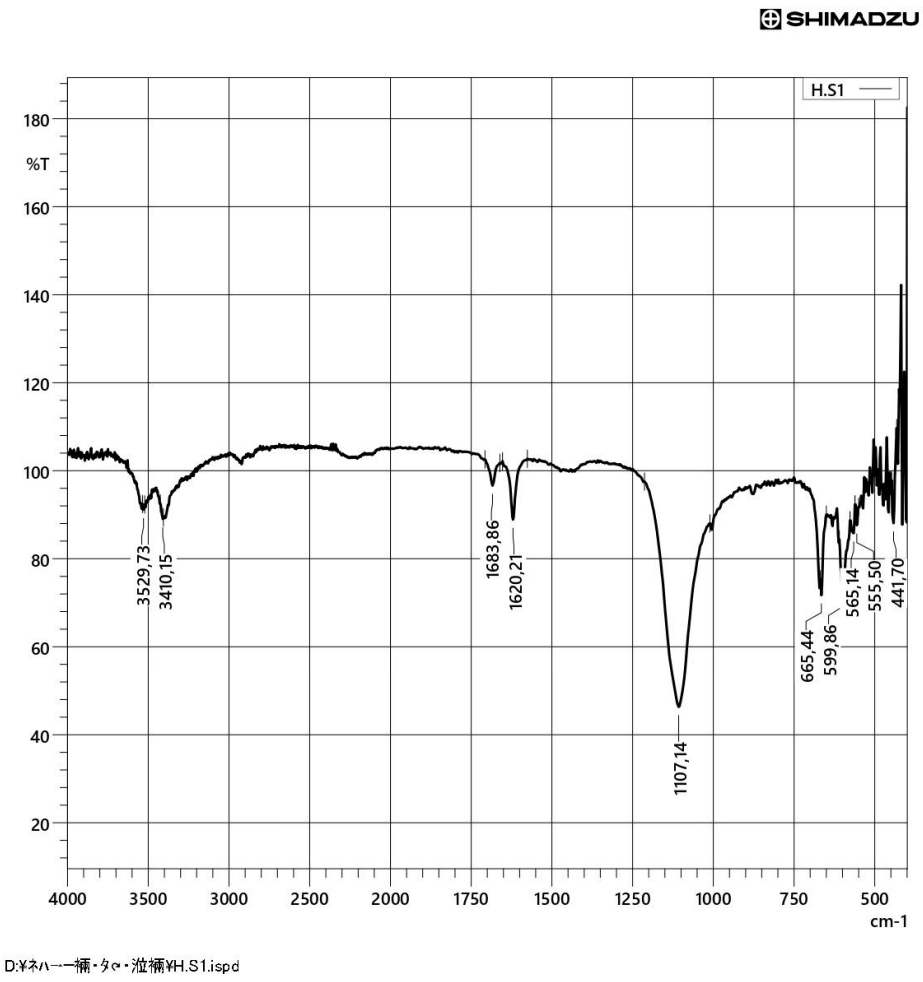
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3	Sample ID	
4	Option	
5	Intensity Mode	%Transmittance
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9	No. of Scans	10

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Figure 6. Infrared spectrum of gypsum concrete without Redispersible Polymer Powder (RPP).

For the unmodified sample, broad absorption bands were observed in the 3000–3700 cm^{-1} region. Absorption regions were also recorded at 2800–3000 cm^{-1}, 1600–1700 cm^{-1}, 1400–1500 cm^{-1}, 1000–1200 cm^{-1} and 500–800 cm^{-1}.

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	Item	Value
2	Sample name	
3	Sample ID	
4	Option	
5	Intensity Mode	%Transmittance
6	Apodization	Happ-Genzel
9	No. of Scans	10

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Figure 7. Infrared spectrum of gypsum concrete containing 0.10% Redispersible Polymer Powder (RPP).

In the sample containing 0.10% RPP, absorption peaks at 3529.73 cm^{-1} and 3410.15 cm^{-1} were recorded in the hydroxyl region. Peaks at 1668.36 cm^{-1} and 1620.21 cm^{-1} were also observed, while the most intensive peak in the 1000–1200 cm^{-1} range was recorded at 1107.14 cm^{-1}. Differences between the spectra of the samples with and without RPP were used to evaluate changes in the physicochemical structure of the gypsum matrix.

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4. Conclusion

1. The physicochemical characteristics of gypsum concrete containing lime waste and 0.10% RPP were studied using thermal analysis, X-ray diffraction, scanning electron microscopy and infrared spectroscopy.

2. In the RPP-modified sample, the main mass loss was recorded in the range of 301.08–601.24 °C, and a pronounced thermal effect was observed at approximately 414.49 °C.

3. X-ray diffraction analysis identified $CaSO_4 \cdot 2H_2O$ as the main crystalline phase and SiO_2 as a secondary phase. The crystallite size of gypsum in the (020) direction was approximately 100 nm.

4. SEM observations showed denser intercrystalline contacts and reduced structural voids in the RPP-modified sample compared with the sample without the polymer additive.

5. Differences in the characteristic infrared absorption regions of the samples with and without RPP were recorded and used to evaluate changes in the physicochemical structure of the gypsum matrix.

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