



EFFECT OF CALCINATION TEMPERATURE ON PHASE TRANSFORMATION AND MICROSTRUCTURAL EVOLUTION OF RED GYPSUM

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ABSTRACT

Red gypsum (RG) is a major byproduct of titanium dioxide production through the sulphate process, and its limited utilization poses significant accumulation challenges. One promising treatment method is thermal modification, particularly calcination, to achieve phase transformation and material stabilization. This study examines the impact of calcination temperatures ranging from 900°C to 1300°C on RG on the phase transformations and structural stability of RG. The raw RG and calcined RG samples were thoroughly characterized using thermogravimetric analysis (TGA), X-ray fluorescence (XRF), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Scanning Electron Microscopy (SEM). TGA results for raw RG revealed weight loss of 19.3% between 100°C and 200°C, attributed to the dehydration of calcium sulphate dihydrate (CaSO₄·2H₂O), followed by a minor weight loss which goethite dihydroxylation between 250°C to 300°C. XRF analysis revealed RG primarily consists of Ca (20-21%), S (11-12%), and Fe (10-11%), with trace amounts of other elements. FTIR spectra of raw RG indicated the presence of H-OH and -OH stretching vibrations, which disappeared after calcination, confirming dehydration. XRD analysis identified CaSO₄·2H₂O and goethite (α-FeOOH) in raw RG, while anhydrite (CaSO₄) and hematite (Fe₂O₃) were dominant after calcination. SEM analysis demonstrated that calcination at 900°C achieved substantial dehydration while limiting excessive sintering, whereas temperatures ≥1200°C resulted in pronounced sintering and structural degradation. Based on phase stability, morphology, and energy efficiency, 900°C is recommended as the optimal calcination temperature. This demonstrates that calcination enhances RG's stability, making it a potentially suitable secondary raw material for use in cement, ceramics, and fertilizers.

Keywords: red gypsum, calcination, thermal stabilization, phase transformation, industrial waste valorization.

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1. INTRODUCTION

Red gypsum (RG) is an industrial byproduct generated during the sulphate-route production of titanium dioxide (TiO₂) [1]. The extraction of titanium from ilmenite ore (FeTiO₃) produces acidic wastewater, which is typically neutralized using calcium carbonate (CaCO₃). While this neutralization process helps mitigate environmental impacts which can generate secondary waste. Specifically, the reaction between sulphuric acid and CaCO₃ produces red gypsum (CaSO₄·2H₂O) and carbon dioxide, as represented in Eq. 1.



Traditionally, RG is disposed of in landfills or stockpiled near titanium dioxide manufacturing facilities. However, recent advances highlight its potential for reuse as a secondary raw material, in line with environmental protection initiatives, sustainable resource utilisation, and the principles of a circular economy. The feasibility of incorporating RG into construction materials has been investigated [2], [3]. Gazquez *et al.* (2013) demonstrated that RG can substitute natural gypsum in Portland cement production without compromising the mechanical integrity

or leaching behaviour of the final product. Similarly, Sotiriadis *et al.* (2024) explored the development of gypsum-cement-pozzolan binders with self-cleaning properties using landfilled RG.

Beyond construction applications, red gypsum has also shown promise in catalysis [4]. Mohd *et al.* (2024) synthesised a heterogeneous catalyst from RG for the production of glycerol carbonate via glycerolysis with urea. Azdarpour *et al.* further evaluated RG as a calcium source for CO₂ sequestration via mineral carbonation, examining the effects of reaction parameters, including CO₂ pressure, temperature, and particle size, on conversion efficiency and product purity [5].

One of the most effective methods for enhancing the properties of RG is calcination, a thermal treatment process that removes water and volatile components, decomposes hydrates, and induces phase transformations [6], [7], [8]. The reddish colouration of RG is attributed to iron impurities and high levels of calcium sulphate dihydrate. Through calcination, RG can be transformed into more stable and functional materials. For instance, Marian *et al.* (2023) utilized RG as a secondary raw material in ceramic production, highlighting its consistent chemical composition and favourable sintering behaviour at



temperatures below 1000°C [9]. Similarly, Mohd et al. (2024) employed calcined RG as a catalyst precursor for glycerol carbonate synthesis, with calcination temperatures of 180°C and 800°C yielding a high product conversion rate of 87% due to improved catalytic activity.

While prior studies have typically employed calcination temperatures below 1000°C [4], [9], [10], such conditions may only achieve partial dehydration and phase modification. In contrast, increasing the calcination temperature above 1000°C may promote further phase transformation and densification. However, excessive heating may also induce sintering and thermal decomposition, and crucial for advanced industrial applications and long-term environmental safety [8], [11]. Therefore, most previous studies calcined red gypsum below 1000°C for catalytic or low-temperature applications; the behaviour at temperature above 1000°C, particularly microstructural degradation and the onset of anhydrite decomposition, remains poorly documented.

Therefore, the objective of this study is to investigate the impact of calcination temperature on the phase transformation and material stabilization of red gypsum. Five temperatures were selected: 900°C, 1000°C, 1100°C, 1200°C, and 1300°C to evaluate the thermal behaviour and material evolution of RG. The results are expected to provide insights into optimizing calcination parameters to enhance material performance and environmental safety. The study hypothesizes that temperatures above 1000°C will facilitate complete dehydration and phase stabilization, albeit at the cost of increased energy consumption.

2. MATERIALS AND METHOD

2.1 Materials

Red gypsum (RG) was obtained from Venator Asia Sdn. Bhd., located in Teluk Kalong, Kemaman, Terengganu, Malaysia. The as-received sample was initially air-dried at ambient conditions ($25 \pm 2^\circ\text{C}$) for 72 hours, followed by oven drying at 80°C for 24 hours to remove residual moisture. The dried RG was ground in a planetary ball mill (PM100, Retsch) to obtain a fine, homogeneous powder. The grinding process was conducted in a 500 mL stainless steel jar using 10 mm stainless steel balls, with a ball-to-powder weight ratio of 10:1. Approximately 20 g of RG was milled at 250 rpm for 1 hour. The resulting powder was sieved through a 63 μm mesh to ensure homogeneous particle size for subsequent analysis.

2.2 Calcination of Red Gypsum

The red gypsum powder was thermally treated via calcination to investigate its thermal behaviour and structural evolution. Calcination was performed using a high-temperature furnace (UNITEX) under atmospheric conditions. For each trial, 50 g of the 63 μm -sized RG powder was placed in an alumina (Al_2O_3) crucible, chosen for its chemical inertness and thermal stability at high

temperatures. The samples were heated at a constant rate of 5°C/min from room temperature ($\sim 25^\circ\text{C}$) to the designated temperatures of 900°C, 1000°C, 1100°C, 1200°C, and 1300°C. Each sample was held at the target temperature for 2 hours to ensure complete thermal transformation and phase equilibrium. After the holding period, the furnace was allowed to cool naturally to room temperature. The calcined samples were stored in airtight containers to prevent moisture reabsorption before characterisation.

2.3 Thermogravimetric Analysis (TGA)

The thermal decomposition profile of raw and calcined RG was evaluated using a TGA/SDTA 851 thermogravimetric analyzer. Approximately 10 mg of sample was placed in a platinum crucible and heated from 0°C to 900°C at 10°C/min under a nitrogen atmosphere (flow rate: 30 mL/min) to suppress oxidation. The weight loss and corresponding temperature data were recorded continuously, and the results were analysed using TA Universal Analysis software to determine decomposition stages and weight-loss percentages.

2.4 X-ray Fluorescence (XRF)

The elemental composition of raw and calcined RG was determined using a handheld Bruker S1 TITAN energy-dispersive X-ray fluorescence (EDXRF) spectrometer. Before analysis, RG powder ($\leq 63 \mu\text{m}$) was loaded into an XRF sample cup and sealed with a polypropylene film to minimize contamination. The device employed a rhodium (Rh) X-ray tube as the excitation source, operating at 15-59 kV and 9.7-18.4 μA . Each sample was scanned for a total of 60 seconds (three 20-second scans) under an air atmosphere using the GeoExploration setting.

2.5 Fourier Transform Infrared (FTIR)

Attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) was used to identify functional groups in the RG samples, using a Bruker Tensor 27 spectrometer equipped with a diamond ATR crystal. Approximately 5 mg of 63 μm RG powder was placed on the ATR crystal and pressed using a pressure clamp to ensure intimate contact. Spectra were recorded over a wavenumber range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} , averaging 32 scans per measurement. Data processing, including baseline correction and peak identification, was performed using OPUS software.

2.6 X-Ray Diffraction

The crystalline phases and structural transformations of RG were analyzed using a Rigaku SmartLab X-ray diffractometer operating at 40 kV and 30 mA. Finely ground RG samples were mounted on sample holders with smooth, compact surfaces to minimise preferred orientation effects. XRD scans were performed over a 2θ range of 10° - 90° with a step size of 0.01° and a scan rate of $5.00^\circ/\text{min}$. Phase identification was conducted



by comparing the obtained patterns with standard reference data.

2.7 Scanning Electron Microscopy

The morphology and microstructure of the raw and calcined RG samples were examined using a TESCAN VEGA scanning electron microscope operated at 15 keV. Before analysis, samples were mounted on aluminium stubs using conductive carbon tape and sputter-coated with gold to approximately 5 nm to enhance conductivity and prevent charging. SEM micrographs were acquired at 2000× magnification to examine detailed crystal structure, surface features, and phase characteristics. The working distance was maintained at 14 mm, and images were captured using a secondary electron (SE) detector.

3. RESULTS AND DISCUSSION

3.1 Physical Observation of RG

Figure-1 illustrates the physical changes in red gypsum (RG) before and after calcination at temperatures ranging from 900°C to 1300°C. Notable transformations in both colour and surface morphology were observed across the temperature range. The original RG sample exhibited a yellowish hue (Figure-1(a)), which progressively darkened with increasing calcination temperatures, eventually turning black at 1300°C (Figure-1(f)). This colour transformation is indicative of phase transitions and thermal decomposition processes occurring during calcination.



Figure-1. Images of Raw RG and Calcined RGs (CRGs) at different temperatures: (a) Raw RG, (b) 900°C CRG, (c) 1000°C CRG, (d) 1100°C CRG, (e) 1200°C CRG, (f) 1300°C CRG.

In addition to colour changes, the particle granularity and surface texture of the samples changed with increased temperature. Higher calcination temperatures led to sintering and agglomeration, resulting in rougher particle surfaces. Above 900°C, sintering became more pronounced as adjacent particles coalesce at contact points, forming larger aggregates. This phenomenon contributed to reduced surface area, altered porosity, and densification, thereby

giving the material a more compact and granular appearance [11].

The observed colour transition from yellow to reddish-brown and ultimately to black is attributed to the thermal decomposition of goethite ($\text{FeO}(\text{OH})$) into hematite (Fe_2O_3), as represented by the reaction: $2\text{FeO}(\text{OH}) \rightarrow \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$ [12]. The transformation of yellowish goethite to reddish hematite contributes to the colour change after heating process [13]. The increasing concentration of Fe_2O_3 at elevated temperatures deepens the red colouration, with excessive Fe_2O_3 and other iron-based impurities ultimately leading to blackening of the sample [14], [15].

3.2 TGA Analysis of Raw RG

Figure-2 presents the TGA/DTG analysis of raw red gypsum (RG) over a temperature range of 25°C to 900°C. A sharp weight loss of 19.3 wt.% is observed between 100 and 200°C, with a strong DTG peak at 148°C. This loss corresponds to the dehydration of calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) to anhydrite (CaSO_4) [14], [15]. The measured loss (19.3 wt.%) is slightly lower than the theoretical value of 20.93 wt.% for pure $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, confirming the presence of minor impurities that are also responsible for the red colour of the material [16].

A small loss of 0.7 wt.% between 250°C and 300°C is due to goethite ($\text{FeO}(\text{OH})$) dihydroxylation to hematite (Fe_2O_3) [15]. A further gradual loss of 2.5 wt.% from 300 to 900°C is attributed to trace carbonate decomposition and the release of residual hydroxyl groups [5], [17]. No significant SO_3 release or substantial sulphate decomposition was detected below 900°C, in agreement with previous studies on red and phosphogypsum [5].

After complete dehydration above 200°C, the resulting anhydrite phase exhibits excellent thermal stability up to 900°C. Therefore, the TGA results confirm that the raw RG is predominantly composed of gypsum with small amounts of iron oxides/hydroxides (goethite/hematite) and carbonates. Following calcination, the material may be suitable for reuse as a secondary raw material in cement production, ceramics, soil stabilization, and environmental applications [18].

Overall, the TGA results confirm that the calcination process effectively removes structural water, facilitates the transformation of thermally unstable phases into more stable mineral forms, and enhances the physicochemical stability of red gypsum. These changes improve its suitability for reuse in industrial applications such as cement production, ceramics, and environmental remediation. The TGA findings indicate that the sample resembles natural gypsum and exhibits strong thermal stability after dehydration, making it suitable for construction and industrial applications.

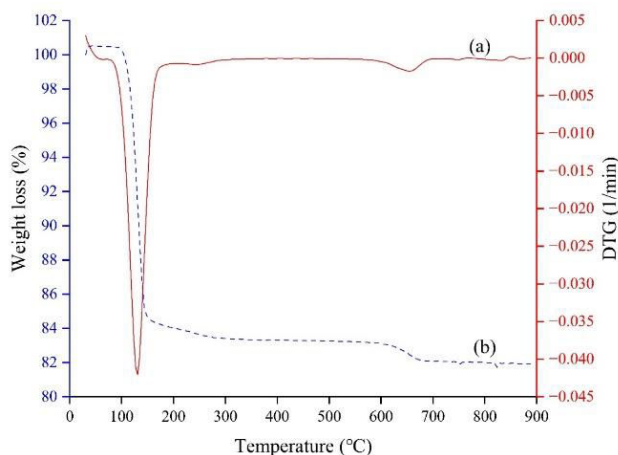


Figure-2. Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) curves of Raw RG (a) weight percentage (%) (b) derivative thermogravimetric (DTG)

3.3 XRF Analysis of RG

The elemental composition of red gypsum (RG) and calcined red gypsum (CRG) at various temperatures, as determined by X-ray fluorescence (XRF) analysis, is presented in Table-1. The results reveal distinct thermal behaviours, reflected in changes in elemental profiles as the calcination temperature increases. The variation in the number of detectable elements with calcination temperature may reflect change in elemental concentration, phase transformation, and the detection limits of the XRF analysis.

The raw RG sample initially contains 24 elemental components. Upon calcination at 900°C, the number decreased to 22. At 1000°C, further decomposition reduces the count to 18 elements, suggesting more extensive

volatilization or chemical breakdown. Interestingly, at 1100°C, the number increased to 22, possibly due to solid-state reactions or the formation of new crystalline phases. At 1200°C, it decreased again to 20, before slightly increasing to 21 at 1300°C, indicating the formation of thermodynamically stable phases. This fluctuation highlights the complex interaction between temperature and elemental stability during thermal treatment.

Across all samples, calcium (Ca) is the dominant element, consistently present at approximately 20-21%, followed by sulphur (S) at 11-12% and iron (Fe) at 10-11% [18]. Trace elements include Ti, SiO₂, Al₂O₃, Mn, P, Cr, V, In, Sr, Zn, K₂O, Zr, Sn, Sb, Cu, Te, Ni, U, Pb, Pd, Y, As, and Th [19]. The Ca content increases from 900°C to 1000°C, decreases at 1100°C, peaks again at 1200°C, and slightly declines at 1300°C, indicating thermal phase and structural changes. The S content remains relatively stable up to 1100°C, followed by a marked decrease at 1200°C and a minor rebound at 1300°C, suggesting thermal decomposition or volatilization of sulphur compounds.

The Fe content shows a non-linear trend, increasing from the raw sample to 900°C, decreasing at 1000°C, and fluctuating thereafter with notable peaks at 900°C and 1300°C. These variations likely reflect redox transformations or phase changes of iron-containing compounds during calcination.

Moreover, the presence of trace elements such as Cr and V is environmentally friendly because of their potential toxicity and should be considered when evaluating the suitability of RG for subsequent applications. These findings illustrate the dynamic thermal behaviour of RG during calcination and provide insights into its elemental stability and transformation mechanisms, which are crucial for evaluating its suitability for high-temperature applications.

**Table-1.** XRF Analysis of Raw RG and Calcined RGs at Different Temperatures.

Element	Composition (%)					
	Raw	900°C	1000°C	1100°C	1200°C	1300°C
Ca	20.370	20.995	21.148	20.699	21.652	20.947
S	12.244	12.346	12.186	12.226	11.695	12.223
Fe	10.137	11.830	10.596	11.640	10.749	11.923
Ti	1.183	1.445	1.253	1.4596	1.243	1.375
SiO ₂	0.414	0.475	0.400	0.387	0.136	0.560
Al ₂ O ₃	0.147	0.380	0.477	0.351	-	0.348
Mn	0.123	0.167	0.147	0.171	0.152	0.176
P	0.031	0.143	0.110	0.139	0.136	0.143
Cr	-	0.031	0.034	0.040	0.036	0.036
V	0.029	0.031	-	0.021	-	-
In	0.018	0.014	0.018	0.022	0.025	0.021
Sr	0.014	0.019	0.018	0.019	0.019	0.018
Zn	0.011	0.015	0.014	0.013	0.013	0.014
K ₂ O	0.011	0.020	0.012	-	0.013	0.025
Zr	0.011	0.010	0.011	0.011	0.012	0.010
Sn	0.011	-	0.007	0.008	0.006	0.008
Sb	0.003	-	-	-	0.004	-
Cu	0.003	0.003	0.004	0.003	0.004	0.004
Te	0.002	0.002	0.001	0.002	0.003	0.002
Ni	-	-	-	0.002	-	-
U	0.002	0.002	0.002	0.002	0.002	0.002
Pb	0.002	0.004	-	0.003	-	0.003
Pd	0.002	-	-	-	-	-
Y	0.001	0.001	0.001	0.001	0.001	0.001
As	0.001	0.001	-	-	-	-
Th	0.001	0.001	-	0.002	0.001	0.001

3.4 FTIR Analysis of RG

Figure-3 presents the FTIR spectra of red gypsum (RG) samples, including raw RG and those calcined at 900°C, 1000°C, 1100°C, 1200°C, and 1300°C. The spectral analysis reveals significant structural changes resulting from calcination. Notably, absorption bands corresponding to hydroxyl (-OH) stretching and H-OH bending vibrations were observed only in the raw RG sample and disappeared after calcination. This confirmed the removal of hydroxyl groups through dehydration during heating.

The raw RG spectrum shows prominent SO₄²⁻ bending vibrations at 600 cm⁻¹ and 669 cm⁻¹, while strong SO₄²⁻ stretching bands are observed at 1115 cm⁻¹ and 1143 cm⁻¹. Additionally, H-OH bending vibrations are evident at 1620 cm⁻¹ and 1682 cm⁻¹, supporting the presence of water molecules in the uncalcined sample. In the region between

2000 and 2500 cm⁻¹, weak absorption peaks are also observed, attributed to SO₄²⁻ vibrations. A marked change is observed in the 3200-3620 cm⁻¹ region, corresponding to the O-H stretching of water. In raw RG, this region shows two intense bands and several weaker peaks and shoulders, which progressively diminish in intensity with increasing calcination temperature. This trend confirms the systematic dehydration of the RG structure and the gradual decline in hydroxyl content with increasing temperature [4], [19].

The spectra of calcined samples at 900°C, 1000°C, 1100°C, 1200°C, and 1300°C exhibited a gradual reduction in overall band intensity, consistent with the thermal breakdown of hydrated phases and the formation of more thermally stable sulphate species. These spectral changes reinforced TGA findings and support the conclusion that



calcination drives significant structural transformations in red gypsum.

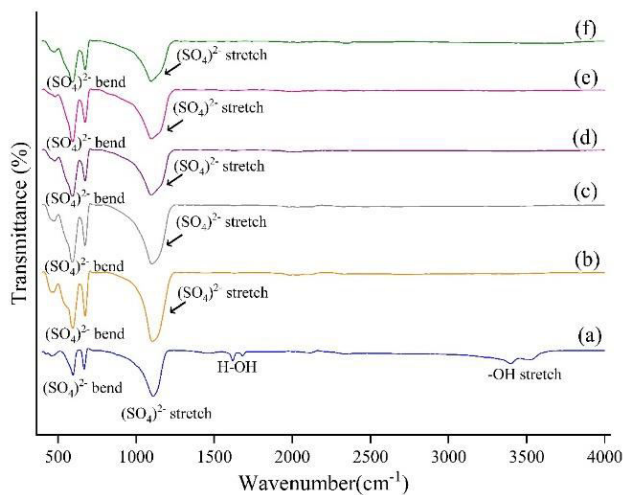


Figure-3. FTIR Analysis of Raw RG and Calcined RG (CRG) at 5 different temperatures: (a) Raw RG, (b) 900°C CRG, (c) 1000°C CRG, (d) 1100°C CRG, (e) 1200°C CRG, (f) 1300°C CRG.

3.5 XRD Analysis of RG

Figure-4 presents the X-ray diffraction (XRD) patterns of red gypsum (RG), showing changes in its crystalline structure and phase composition across various calcination temperatures. The analysis was conducted over a 2θ range of 10° to 90° at a scan speed of $5^\circ/\text{min}$.

In the raw RG sample (Figure-4(a)), two primary crystalline phases are identified: calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and goethite ($\alpha\text{-FeOOH}$). Upon calcination, notable phase transformations are observed. In the samples calcined at 900°C (b), 1000°C (c), 1100°C (d), 1200°C (e), and 1300°C (f), the crystalline phases shifted to anhydrite (CaSO_4) and hematite (Fe_2O_3).

The diffraction peak intensities of hematite increased progressively with rising calcination temperature, indicating enhanced crystallinity and phase transformation at elevated temperatures. This trend suggests that thermal treatment promotes the decomposition of hydrated calcium sulphate and the formation of stable iron oxide phases. The increasing dominance of hematite at higher temperatures was associated with the iron-containing phase observed in the sample [17].

These findings are in close agreement with the XRD patterns reported by Mohd et al. (2024), further confirming the thermal decomposition behaviour and crystallographic evolution of red gypsum during calcination.

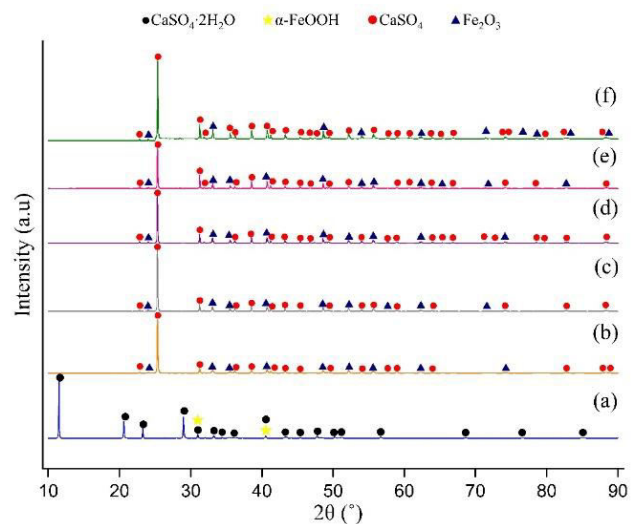


Figure-4. XRD Analysis of Raw RG and Calcined RG (CRG) at 5 different temperatures: (a) Raw RG, (b) 900°C CRG, (c) 1000°C CRG, (d) 1100°C CRG, (e) 1200°C CRG, (f) 1300°C CRG.

3.6 SEM Analysis of RG

Figure-5 presents higher-magnification ($2000\times$) SEM micrographs that reveal detailed particle-level morphological changes in red gypsum during calcination. The raw RG consists of irregular, plate-like gypsum crystals with rough surfaces and a broad particle-size distribution, typical of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ [7], [20]. After calcination at 900°C, dehydration was complete, and the material comprises fractured, angular anhydrite particles with increased surface roughness and microcracks resulting from the escape of crystallization water [21]. At 1000°C, recrystallization of anhydrite II leads to more defined, granular particles and reduced inter-particle adhesion. At 1100°C, sintering became evident through particle rounding and the formation of inter-particle necks, while the anhydrite phase remains dominant. At 1200°C, extensive sintering and particle fusion occur, accompanied by a reduction in surface irregularities. The smoother surface and reduced porosity indicate enhanced particle coalescence and densification at elevated temperature [22]. At a peak temperature of 1300°C, decomposition and particle fusion became more pronounced, producing densely fused irregular aggregates with large voids, and crater-like surface fractures, consistent with advanced thermal alteration and volume contraction [23].

These high-magnification observations further demonstrate the progressive morphological changes induced by calcination. Calcination temperature up to 1100°C yields progressively sintered anhydrite with a gradually decreasing specific surface area, whereas temperatures of 1200°C promote significant sintering, thermal decomposition, and structural changes at elevated temperatures. This information is essential for tailoring calcination temperature to achieve the desired phase composition, microstructure, and reactivity for specific applications.

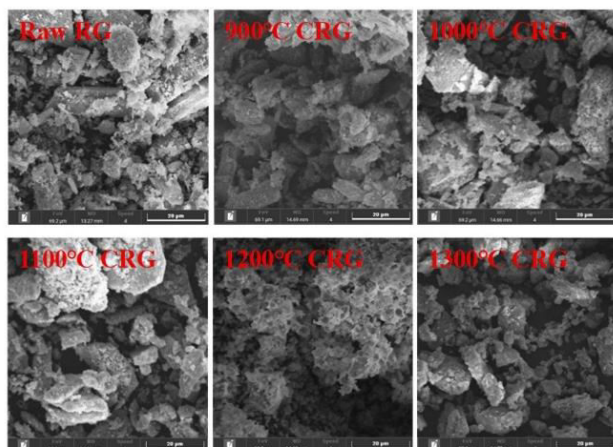


Figure-5. SEM micrographs at high magnification (2000×) for morphological evolution of Raw RG and Calcined RG (CRG) at 5 different temperatures.

4. CONCLUSIONS

The phase transformation and compositional behaviour of red gypsum (RG) were strongly influenced by calcination temperatures ranging from 900°C to 1300°C. At 900°C, RG underwent partial thermal transformation, with key elemental constituents such as Ca, S, and Fe, as well as trace elements including Ti, SiO₂, Al₂O₃, Mn, P, Cr, V, In, Sr, Zn, K₂O, Zr, Cu, Te, U, Pb, Y, As, and Th, confirmed by X-ray fluorescence (XRF) analysis.

During calcination, calcium sulphate dihydrate (CaSO₄·2H₂O) decomposes, releasing water and forming calcium sulphate anhydrite (CaSO₄). Simultaneously, hematite (Fe₂O₃) formation increases significantly with increasing temperature, contributing to the deep red colouration of the calcined samples.

As the temperature exceeds 1000°C, further phase transformation and densification occurred. However, excessive heating also promoted sintering and thermal decomposition.

Based on the observed phase transformation and microstructural change, 900°C provided substantial dehydration while limiting excessive sintering and thermal degradation. This temperature provided sufficient structural and chemical transformation of red gypsum while reducing the thermal energy requirement compared with higher calcination temperatures, making it a practical and sustainable choice for red gypsum treatment in future industrial and environmental applications.

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