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Characterization and Process Optimization of Microwave Drying of Plaster of Paris

Magesh Ganesapillai,[1] Iyyaswami Regupathi,[2] and Thanapalan Murugesan[3]
[1]*Department of Chemical Engineering, A.C. College of Technology, Anna University, Chennai, India*
[2]*Department of Chemical Engineering, National Institute of Technology, Karnataka, Mangalore, India*
[3]*Chemical Engineering Programme, Universiti Teknologi PETRONAS, Bandar Seri Iskandar, Tronoh, Perak, Malaysia*

The changes in the characteristics of plaster of Paris (pop) during drying operation under microwave irradiation conditions, namely surface morphology, effective moisture diffusivity, and absorption of microwave, were studied. The drying characteristics and kinetics of the process during microwave drying of plaster were studied for rectangular-faced cuboids (80 × 70 × 15, L × B × H in mm) through various drying parameters like microwave power input, initial moisture content, and drying time. Further, the experimental data on moisture ratio of plaster for different operating conditions were obtained and the optimization of the microwave drying process parameters was performed with response surface methodology (RSM) by considering all the above-said independent variables. Based on the RSM analysis, the optimum values of the process variables were obtained as: initial moisture content (A) 60%; microwave power input (B) 180 W; and drying time (C) 480 S.

Keywords Characterization; Microwave drying; Optimization; Plaster of Paris

INTRODUCTION

Commercial plaster of Paris (POP; calcium sulfate hemihydrate as its main compound $CaSO_{4\cdot 1/2}H_2O$) is manufactured by the partial dehydration of gypsum. When the dry plaster powder is mixed with water, it reforms into gypsum, initially as a paste but eventually hardened into a solid, and develops strength. The heat energy supplied to gypsum at this condition (the heat of hydration) tends to drive off water (as water vapor) rather than increasing the temperature of the mineral. The hardened mass is not a compact solid but a highly porous material with a relatively large internal surface area consisting of interlocking crystals in the form of plates and needles. The structure consists of sheets of Ca^{2+} and $SO_4{}^{2-}$ ions held together by hydrogen bonds in the water molecules. The microstructure of hardened gypsum paste affects most of the physical and engineering properties, particularly its rigidity. Plaster of Paris, based on the porosity and strength, is widely used as fire resistance on residential and other structures, casted into various shapes including sheets, sticks, and molds (to immobilize metal casting), plaster bandages and strips of cheese (cloth packed with plaster). Small amounts of calcined gypsum are added to earth to create strong structures directly from cast earth, an alternative to adobe (which loses its strength when it contains moisture). The morphology of plaster crystals depends on the formation conditions and the drying methodology put into practice. The nature of moisture removal can be changed to adjust the porosity of the hemihydrates, resulting in the formation of alpha and beta hemihydrates, which are chemically identical.[1] Several drying methods are commercially available and the selection of the optimal method for drying is determined by quality requirements, raw material characteristics, and economic factors. Industrial and geochemical interest has resulted in a considerable amount of research on the mechanism of plaster crystal growth and the nature of drying technique adopted.

A wide range of conventional and nonconventional technologies have been developed in the past for drying of nonconductive and porous solids. These techniques include tray drying,[2] direct solar drying,[3] open air sun drying,[4] and single-layer solar drying.[5–7] The conventionally dried plaster is often difficult to rehydrate because of case hardening and shrinkage during the process. Major disadvantages of convective drying are low energy efficiency and long duration due to the limited heat transfer rate to the inner sections of plaster. In recent years, microwave drying offered an alternative technique, to improve the quality of dehydrated products due to its reduced contact time. Microwaves are electromagnetic waves at

Correspondence: Thanapalan Murugesan, Chemical Engineering Programme, Universiti Teknologi Petronas, Bandar Seri Iskandar 31750, Tronoh, Perak, Malaysia; E-mail: murugesan@petronas.com.my

frequencies between 300 MHz and 300 GHz that propagate through the materials, and the accompanying transport processes result in dissipation of electric energy into heat, which lead to the term ''volumetric heat generation'' due to microwave radiations. While microwave is progressing, the energy radiates from a source in all directions, while the material absorbs this energy and converts it to heat by polar molecules. Water molecules convert microwave energy to heat; when it starts to evaporate, as a result of this heat, the material starts drying.[8] In microwave heating, regions of higher moisture content absorb more microwave energy, where heat is generated throughout the material, leading to faster heating rates and shorter processing times. The effective moisture diffusivity could be described by all possible mechanism of mass transfer within the material, such as liquid diffusion, vapor diffusion, surface diffusion, capillary flow, and hydrodynamic flow. The effective moisture diffusion coefficient depends on the conditions within the material, like moisture content, physical structure of the product, temperature, etc. Other advantages of microwave include space savings and energy efficiency. If suitably applied, microwave drying is faster and probably cheaper on a long-term basis after an initial capital cost outlay for equipment. In recent years, microwave energy has found increasing application for thermal processing of various kinds of materials and products in the areas of agricultural,[9–12] chemical,[13–15] ceramic,[16–18] mineral,[19–21] food,[22–25] leather,[26] pharmaceutical,[27,28] etc.

The objective of the present research is to dry (molded/tempered) plaster with different levels of initial moisture content in microwave atmosphere and to study the rate of moisture removal as drying rate and to study the raise in temperature of the molded plaster in the presence of carrier water and the effective moisture diffusivity coefficient of plaster. Further, the optimization of experimental variables is carried out using response surface methodology (RSM). A set of experimental condition is developed for the microwave drying process to perform the RSM, using central composite design (CCD). In this work, the effect of drying time, initial moisture content of sample, and input power during exposure were assessed through RSM for the moisture removal and optimization of the process.

METHODS AND MATERIALS

Sample Preparation

High-purity, analar grade plaster with a particle size less than 50 μm was purchased from SRL, India. Rectangular-faced cuboids ($80 \times 70 \times 15$, $L \times B \times H$ in mm) were molded by mixing plaster with deionized water (carrier) in the ratio 3:2. Slip casting was undertaken at normal ambient temperature and were initially tempered for a period of 18 h, which allows the material to become rigid and stable.[29] The samples thus prepared with an initial moisture content of 60–90% were further used for microwave drying experiments.

Microwave Experimental Setup and Procedure

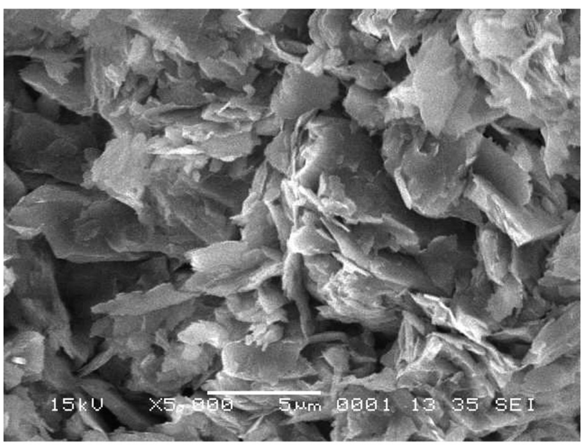
A programmable domestic microwave oven (SAMSUNG C-103F model) having an inbuilt bio-ceramic cavity ($352 \times 300 \times 220$, $L \times B \times H$ in mm) with a maximum power output of 900 W and a frequency of 2450 MHz was used. The system was operated at three levels of output microwave power, viz. 540, 360, and 180 W, corresponding to 60, 40, and 20% of its maximum power. An inbuilt weighing system was used to monitor the weight loss of the sample continuously.[30] A circular hole of 15 mm in diameter was cut at the base of the microwave oven chamber, through which a weighing beam (quartz made) was introduced to hold the sample at its top. The bottom of the beam was placed in the load cell (sensitivity ± 0.01 g) to measure the load placed in the top of the weighing beam, which was connected to the digital converter through a weighing recorder and a voltmeter. The uninterrupted mass change measurement was achieved by drilling three 3.2-mm-diameter holes in the bottom of the test chamber. Teflon pins of 3 mm diameter were attached to a platform resting on the electronic balance passing through the holes. The sample container sat on the Teflon pins, thus exerting its weight directly on the balance. The arrangements enabled online measurement of the sample weight on the balance by diverting the microwave supply into the atmosphere temporarily for few seconds while measuring the weight of the sample. The balance took about 5 s to stabilize. The oven was provided with a magnetron for producing dielectric waves, a fluorescent bulb, and a circulating fan for the distribution of the produced microwaves. Proper microwave leak-proof agents were provided at the bottom of the cavity where the hole was made.

A series of drying experiments was performed with samples containing moisture content of 60–90% (wet basis) in the capillary pores. Microwave drying of tempered plaster was continued for a desired period of time with constant power input. All experiments were conducted at three different initial moisture contents (approximately 60, 75, and 90%) and three power inputs (540, 360, and 180 W). The mass of the samples used for the experiments was approximately 150 g. To study the influence of sample thickness on effective moisture diffusivity the experiments were conducted for various sample thicknesses in a range of 11–15 mm for a constant microwave power input of 180 W with an initial moisture of 90%. All experiments were performed in triplicate and the average values were used for further analysis of data on drying rate and moisture removal.

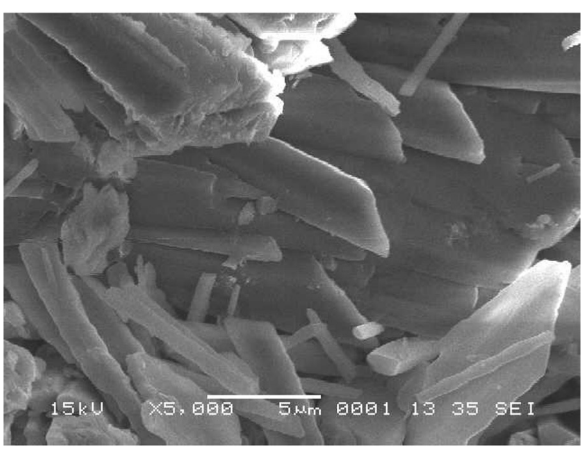
Characterization of Plaster and Water

Surface Morphology of Microwave Activated Plaster

Figures 1a and 1b show the scanning electron microscope (SEM) images of the precursor (plaster) and the microwave-dried plaster, respectively. It is apparent from Fig. 1a that there were very few pores available on the surface of the precursor and the crystalline nature of the components was not clear. However, after microwave drying (microwave input power of and initial moisture content of sample 90%, for a period of 150 s), large size and a greater number of pores were developed on the surface of the microwave-irradiated plaster in addition to the crystal formation, as shown in Fig. 1b. This leads to the conclusion that microwave drying was effective in creating well-developed pores on the surfaces of the precursor; hence, the microwave-irradiated plaster contains a large surface area and porous structure, which has a high drying rate with minimum exposure. Similar observations were reported by other researchers in their work using jute and coconut fibers.[31]



(a)



(b)

FIG. 1. (a) SEM images of plaster of Paris before microwave irradiation; (b) SEM images of plaster of Paris after microwave irradiation.

Absorption Characteristics of Plaster and Water

Polar substances absorb microwaves to convert microwave energy to thermal energy, in which the rate of absorption is determined by the dipole rotation and ionic conductance characteristics of the material. The water molecules (dipolar substance) present in molded plaster provide heating by dipole rotation. The magnitude of thermal energy deposited into materials depends on the internal electric field strength, the frequency of the microwave radiation, and the dielectric properties of the material.[32] The primitive equation to calculate the power absorption density per unit volume for dipolar rotation can be approximated, if the electric field strength is

$$P_V = kE^2 f \varepsilon' \tan\delta \tag{1}$$

or

$$P_V = kE^2 f \varepsilon'' \tag{2}$$

where P_V is the power density (W/m^3), k is a constant, E is the electric field strength (V/m), f is the microwave frequency (2450 MHz), $\tan\delta$ is the dissipation factor, ε' (the ability to store electrical energy) and ε'' (amount of energy a dielectric material can dissipate in the form of heat) are the dielectric constants, which are a measure of the charge retention capacity of a medium and dielectric loss (the imaginary part of the dielectric constant, which determines the lossiness of the medium), respectively. The following equations were used to measure the complex permittivity were the sample material does not have significant magnetic properties.

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \tag{3}$$

$$\tan\delta = \left[\frac{\varepsilon''}{\varepsilon'}\right] \tag{4}$$

where ε^* is the complex permittivity (the ability of material to couple electrical energy from a microwave field), $j = (-1)^{1/2}$, and $\tan\delta$ is the loss tangent (the ratio of dielectric loss to dielectric constant). To enlighten the influence of microwave heating, the specific heat, thermal conductivity, and dielectric properties of materials were considered[33,34] (Table 1). At a fixed microwave frequency, only the material properties ε^* and $\tan\delta$ determine the total power dissipated within the sample.[33–35] Further, both the parameters depend strongly on temperature. To study the interaction characteristics of plaster with water, raw plaster and water were individually spread on the Teflon-lined sheet, placed into a microwave oven, and then irradiated at three power levels. Then the experimental time versus temperature curves for water, raw plaster, and molded plaster were plotted for increasing power inputs in microwave system.

Effective Moisture Diffusivity

The intrinsic mass transfer property of any material is determined by its effective moisture diffusivity. The

TABLE 1
The thermal and dielectric properties of plaster of Paris and water[33,34]

S. no.	Material properties	Water	Plaster
1	Density, ρ (kgm^{-3})	1000	2300
2	Thermal conductivity k ($Wm^{-1}K^{-1}$)	0.609	0.480
3	Heat capacity Cp ($Jkg^{-1}K^{-1}$)	4190	9000
4	Dielectric constant ε' (at 2450 MHz)	78.1	2.5–6.0
5	Dielectric loss ε'' (at 2450 MHz)	10.44	—

mechanism of movement (removal) of moisture from the interior to the surface of plaster during the microwave operation was exclusively due to diffusivity as explained by Fick's second law. The effective moisture diffusivity purely depends on the composition of the sample, moisture retained, and the temperature attained during the process apart from the porosity of the material. Molded and tempered rectangular plaster cuboids were assumed as a slab and the following assumptions were made: Initial moisture distribution is uniform throughout the mass of a sample; mass transfer is symmetric with respect to the center; surface moisture content of the sample instantaneously reaches equilibrium with the condition of surrounding air; resistance to mass transfer at the surface is negligible compared to internal resistance of the sample; diffusion coefficient is constant and shrinkage is negligible. Based on these assumptions, the effective moisture diffusivity was calculated, as expressed by Crank:[36]

$$MR = \left[\frac{M_t - M_e}{M_0 - M_e}\right] = \frac{8}{\pi^2}\exp\left[-\frac{(D_{eff}\cdot\pi^2)}{T^2}t\right] \tag{5}$$

where M_t is the moisture content at any specific time (g/g (dry basis)), M_0 is the initial moisture content (g/g (dry basis)), M_e is the equilibrium moisture content (g/g (dry basis)), D_{eff} is the effective moisture diffusivity ($m^2{\cdot}s^{-1}$), T is the half thickness of plaster of Paris ($T = 0.0075$ m), and t is the drying time (s). Several researchers demonstrated that the above equation could be simplified to a linear equation[37–39] as given below:

$$\ln(MR) = \ln\left[\frac{8}{\pi^2}\right] - \left[\frac{(D_{eff}\cdot\pi^2)}{T^2}\right]\cdot t \tag{6}$$

The effective moisture diffusivities are typically determined by plotting the linear relationship between ln(MR) and drying time at various microwave output powers and initial moisture content of plaster of Paris.

Optimization through RSM

During preliminary experiments, the various initial moisture content of plaster, time of exposure of sample to microwave, microwave power input, surface area, and geometries for their suitability to sustain maximum drying (moisture removal) of plaster by microwave were evaluated. Preliminary analysis of data indicated that the major variables affecting the drying rate of the sample are moisture content of sample, power input to the system, and drying time. Therefore, these three parameters were chosen for further optimization through RSM.[40–43] The application of statistical experimental design techniques in drying process development can result in improved product yields, reduced process variability, closer confirmation of the output response to nominal, targeted requirements, and reduced development time and overall costs. In order to describe the nature of the response surface in the experimental region and elucidate the optimal conditions of the most significant independent variables, the RSM was applied for the current data on microwave drying process and was extended to analyze the interaction between variables.

A three-level, three-factor central composite design (CCD) was performed using the software Minitab 15 (Minitab Inc.). The independent variables like initial moisture content (A), microwave power input (B), and drying time (C) were chosen for the removal of moisture content in plaster. The actual factor levels examined at three levels (low, basal, and high), corresponding to the coded factor levels (−1, 0, +1), are shown in Table 2. The upper and the lower limits of each variable were chosen by means of the characteristic of plaster and the preliminary investigations. Generally, the CCD consists of a 2^n factorial runs (n = 3), with $2n$ axial runs and n_c center runs (six replicates); the total number of scouring experiments was altogether 20, as calculated from Eq. (7):

$$N = 2^n + 2n + n_c = 2^3 + 2\times 3 + 6 = 20 \tag{7}$$

where N is the total number of experiments required and n is the number of factors (3). Based on Table 2, the experiments were conducted to obtain the response; i.e., rate of drying of plaster is carried out at the corresponding independent variables addressed in the experimental design matrix. A second-order polynomial equation was considered to represent the present experimental data.

$$Y_i = \beta_0 + \sum_i \beta_i x_i + \sum_{ii} \beta_{ii} x_i^2 + \sum_{ij} \beta_{ij} x_i x_j \tag{8}$$

where Y_i is the predicted response, $x_i x_j$ are the independent variables, β_i is the ith linear variable coefficient, β_{ij} is the ijth interaction coefficient, and the independent

TABLE 2
Independent variables for the microwave drying of plaster of Paris by central composite design (CCD) and actual and predicted response

Run order	Coded variable x_1	x_2	x_3	Actual variable *A*: Initial moisture content (%)	*B*: Microwave power input (W)	*C*: Drying time (s)	*Y*: Response Actual	Predicted
1	0	0	0	75	360	300	67.63	67.46
2	0	−1	−1	75	360	180	91.85	92.86
3	−1	−1	0	60	360	300	73.50	73.38
4	0	−1	0	75	540	300	55.66	55.73
5	1	1	1	90	540	420	54.86	55.46
6	0	0	0	75	360	300	68.21	67.46
7	0	0	0	75	360	300	67.21	67.46
8	0	0	0	75	360	300	67.52	67.46
9	0	−1	0	75	180	300	36.23	37.25
10	0	−1	1	75	360	420	68.52	68.61
11	0	0	0	75	360	300	68.21	67.46
12	−1	1	1	60	540	420	67.25	66.83
13	1	1	−1	90	180	180	64.81	64.95
14	0	0	0	75	360	300	68.21	67.46
15	1	1	1	90	180	420	30.82	29.84
16	−1	1	−1	60	180	180	69.75	68.87
17	−1	1	−1	60	540	180	79.52	80.22
18	−1	1	1	60	180	420	46.09	46.79
19	1	1	−1	90	540	180	82.86	81.88
20	1	0	0	90	360	300	64.52	65.73

variables were *A*, *B*, and *C*. In this study, a second-order polynomial equation can be delivered as such:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC \quad (9)$$

where Y is the dependent variable (drying rate); *A*, *B*, and *C* are the independent variables as mentioned above; β_0 is the regression coefficient at center point; β_1, β_2, and β_3 are linear coefficients; β_{12}, β_{13}, and β_{23} are second-order interaction coefficients; and β_{11}, β_{22}, and β_{33} are quadratic coefficients. The values of the coefficients, the optimum levels, as well as R^2 were determined as mentioned above. Response surface graphs were plotted for all experimental data by the regression analysis using the statistical software. The statistical parameters were examined with analysis of variance (ANOVA). The implications of the model equations and terms were evaluated by Fischer's test. The quality of fit of the polynomial model equation was expressed by the coefficient of determination (R^2), adjusted R^2, and "adequate precision." The fitted polynomial equation was expressed as three-dimensional surface plots to visualize the relationship between the responses and the experimental levels of each factor used in the design. The combination of different optimized parameters, which gave maximum response, i.e., maximum drying (moisture removal), was tested experimentally to validate the model.

RESULTS AND DISCUSSION

Microwave Adsorption Characteristics

Plaster of Paris is a comparatively poor microwave absorber, since the carrier (water) used to bind is an excellent absorber, a quick warming up of the sample has occurred. When water alone was placed in the microwave oven, the temperature of water molecules increased rapidly and the boiling point was attained in 300 s at a minimum input power (180 W), while the same conditions were reached before 180 s at 540 W (Fig. 2). The temperature of raw plaster increased gradually to a maximum of 42°C by 300 s when the input power was maintained around 180 W, whereas for the same duration of exposure of plaster, a maximum temperature of 35°C was obtained at 540 W as shown in Fig. 3. In the case of molded plaster, a few minutes of exposure to microwave irradiation even at low power levels raises the temperature of the slab above 50°C. The samples processed for 300 s at 180, 360, and

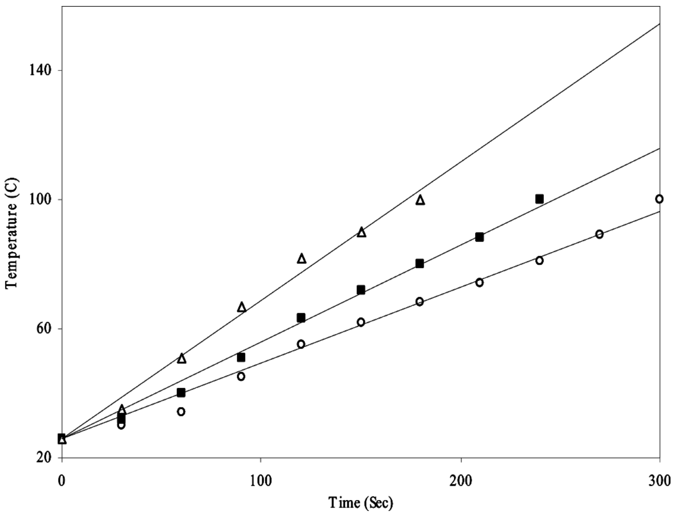


FIG. 2. Effect of power dependent microwave heating of water: ○ 180 W: ■ 360 W: Δ 540 W.

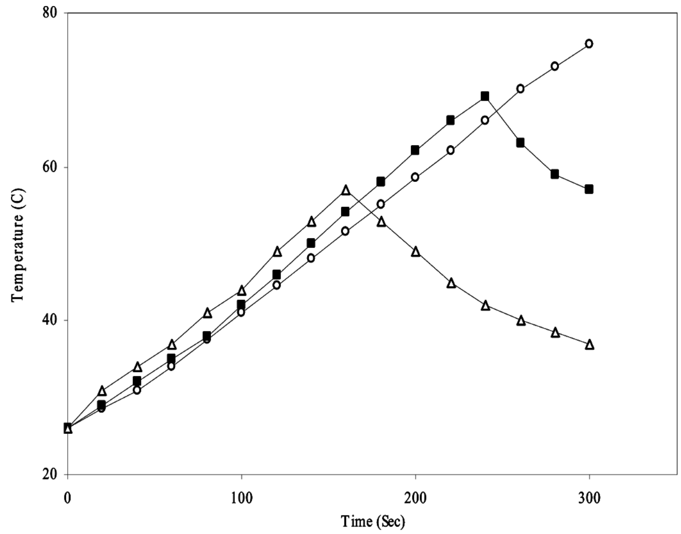


FIG. 4. Effect of power dependent microwave heating of water on plaster of Paris: ○ 180 W: ■ 360 W: Δ 540 W.

540 W showed a significant raise in the temperature, thus a maximum of 76°C was achieved at 180 W (Fig. 4). This strategy may be advantageous for achieving a greater heating rate because when water molecules that convert microwave energy to heat start to evaporate, the material starts drying. In microwave heating, regions of higher moisture content absorb more microwave energy, where heat is generated throughout the material. Thermal runaway, a complicated phenomenon occurs due to the size and geometry of samples, dielectric properties, etc., is highly absent in this case, which normally takes a major role and hinders the complete process.[33] According to the present study, drying of plaster begins approximately 20 s after the operation commences, and a steady-state conversion is attained. As seen from Fig. 4, in microwave heating complete drying of plaster was achieved within 300 s at 180 W. Moreover, microwave heating provides great time savings, compared with conventional drying.

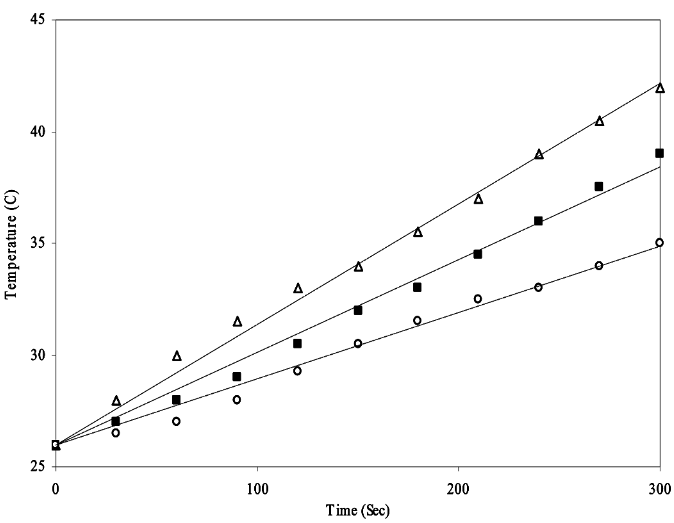


FIG. 3. Effect of power dependent microwave heating of raw plaster of Paris: Δ 180 W: ■ 360 W: ○ 540 W.

X-ray diffraction (XRD) patterns of the samples are shown in Figs. 5a and 5b. X-ray diffraction indicates the geometry or shape of a material using X-rays and is based on the elastic scattering of X-rays from structures that have a long range order. Powder X-ray diffraction of sample essentially shows peaks corresponding to calcium sulphate in Fig. 5a, whereas in Fig. 5b the powder X-ray diffraction of microwave-irradiated product essentially shows peaks corresponding to calcium sulphate with large number of peaks and more d-spacings at 2 theta, indicating the scattered pattern during the interaction of water molecules with crystals of calcium sulphate.

Effective Moisture Diffusivity during Microwave Drying of Plaster

The effective diffusivity includes the total movement of water through liquid diffusion, vapor diffusion, vaporization-condensation, hydrodynamic flow, and other possible mass transfer properties of water in the material. The slopes of the graph, drawn between the logarithm value of moisture ratio and drying time for different microwave output powers ranging from 540 to 180 W, represent the effective diffusivity of plaster of Paris during the drying process. The effective moisture diffusivity values (D_{eff}) in Eq. (6) are shown in Table 3 for different levels of microwave power inputs and moisture content. The samples with higher moisture content responded quantitatively large to microwaves, resulting in fast and complete drying of

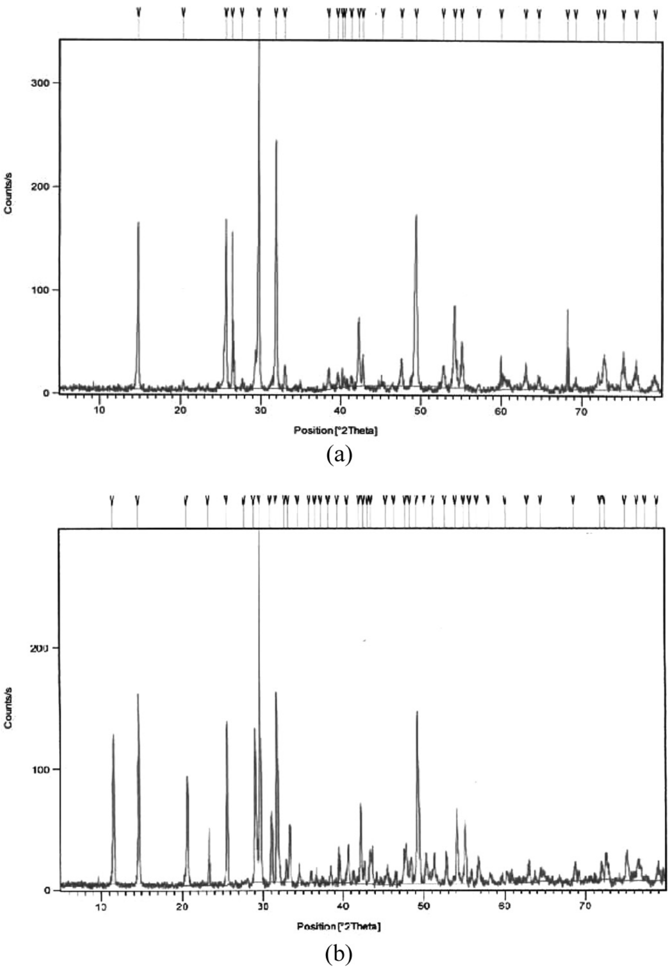


FIG. 5. (a) XRD pattern of plaster of Paris before microwave irradiation; (b) XRD pattern of plaster of Paris after microwave irradiation.

plaster, whereas the process was slow for samples with less moisture content.[44] For a maximum initial moisture of 90% and lowest power of 180 W, the effective moisture diffusivity was estimated as 2.0382×10^{-8}($m^2 \cdot s^{-1}$), whereas for a lowest initial moisture content of 60% and maximum power input of 540 W, the effective moisture diffusivity was found to be 0.8215×10^{-8} ($m^2 \cdot s^{-1}$). The effect of drying time on the logarithmic value of moisture ratio is illustrated in Fig. 6 for all microwave powers and initial moisture contents.

It was observed from the experiments that, at high power (540 W) input levels, the continuous supply of microwave energy produces the sample temperature to attain a point in which the samples begin to crack, while lower power favors maximum drying of POP. Since the solvent used in the sample was water, which is dipolar in nature, the water molecules absorb the microwave instantly and rapidly. A fast rate and complete drying of POP were accomplished for samples with maximum initial moisture content, whereas comparatively slower rates were observed for cases where the initial moisture content was less.

Further, the effect of sample thickness on the effective diffusivity for the microwave drying of plaster was studied by conducting experiments for various thicknesses of the plaster ranging from 11 to 15 mm, at a constant microwave power level of 180 W and initial moisture content of 90%. It was observed from Fig. 7 that the relation between ln (MR) and drying time was linear; the drying time was proportional to sample thickness, while the drying rate and effective moisture diffusivity were inversely proportional. The coefficients of linear model (Eq. (6)), at various thicknesses of plaster of Paris, the effective moisture diffusivity (D_{eff}), and the corresponding values of coefficients of determination R^2 are presented in Table 4 for each sample thicknesses. For samples of less thickness, as a result of high energy transferred to the material, the vapor pressure inside the product increased, leading to faster diffusion of moisture to the surface.[45] The effective moisture diffusivity for samples remained at 2.0382×10^{-8} ($m^2 \cdot s^{-1}$) for the 15-mm samples, whereas it increased to 4.764×10^{-8} ($m^2 \cdot s^{-1}$) for a lowest sample thickness of 11 mm. As the

TABLE 3
Coefficients of linear model in Eq. (6) at various microwave output powers and initial moisture content for plaster of Paris (sample thickness 0.015 m)

S. no.	Moisture content	Power input	Slope $\times 10^3$	R^2	$D_{eff} \times 10^8 (m^2 \cdot s^{-1})$
1	90	180	3.579	0.9949	2.0382
2	90	360	3.310	0.9938	1.8851
3	75	180	2.766	0.9934	1.5749
4	60	180	2.331	0.9916	1.3276
5	75	360	2.151	0.9910	1.2243
6	75	540	1.887	0.9887	1.0743
7	90	540	1.737	0.9858	0.9890
8	60	360	1.522	0.9701	0.8669
9	60	540	1.443	0.9502	0.8215

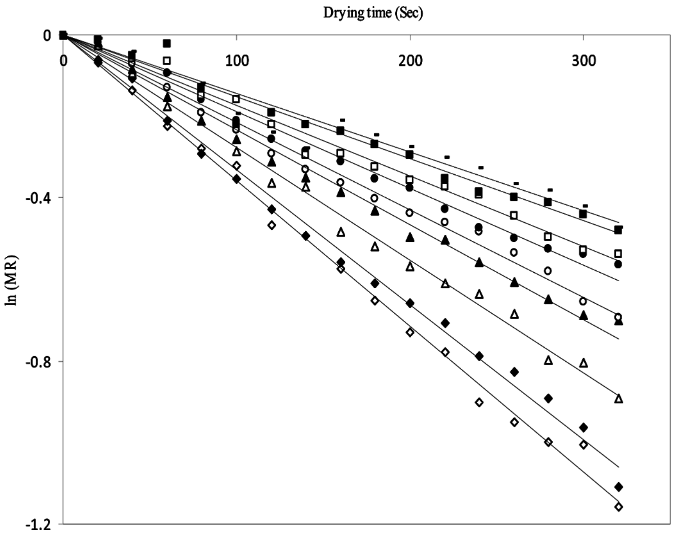


FIG. 6. Drying kinetic relationship of POP at different microwave power ranges for 15-mm sample thickness: ◇ 180 W, IM 90%; ◆ 360 W, IM 90%; Δ 180 W, IM 75%; ▲ 180 W, IM 60%; ○ 360 W, IM 75%; ● 540 W, IM 75%; □ 540 W, IM 90%; ■ 360 W, IM 60%; – 540 W, IM 60%.

drying process proceeded, the loss of water in the sample caused a decrease in the absorption of microwave power to the sample and drying rate started to decrease; moreover, as the sample thickness was increased, the drying rate apparently decreased.

Drying Kinetics of Plaster

The microwave power supplied to the system plays a major role to arrive at a desired final moisture content,

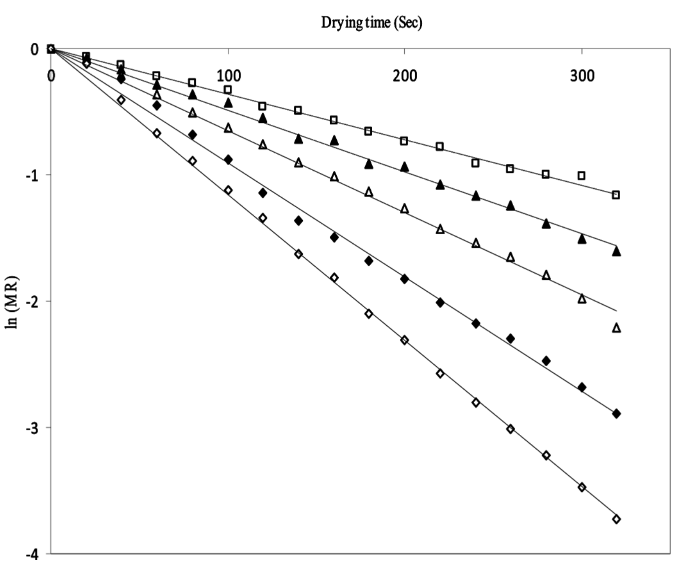


FIG. 7. Drying kinetic relationship of POP at varied thickness of plaster of Paris (microwave input power 180 W, IM 90%): ◇ 15 mm; ◆ 14 mm; Δ 13 mm; ▲ 12 mm; □ 11 mm.

TABLE 4
Coefficients of linear model in Eq. (6) at various thicknesses of plaster of Paris (microwave output powers: 180 W, initial moisture content 90%)

S. no.	Sample thickness (m)	Slope × 10^3	R^2	$D_{eff} \times 10^8 (m^2 \cdot s^{-1})$
1	0.015	3.59	0.9950	2.044
2	0.014	4.88	0.9955	2.421
3	0.013	6.50	0.9957	2.779
4	0.012	9.02	0.996	3.288
5	0.011	15.5	0.9990	4.746

which explains the drying nature of samples. During the experiments, it was observed that at high input power (540 W), the continuous supply of microwave energy increases the sample temperature at which the samples begin to crack. Hence, operation at lower power was favorable for complete drying of plaster. A fast rate and complete drying of plaster were accomplished for samples that had maximum initial moisture content, whereas comparatively slower rates were observed for cases where the initial moisture content was less. Since the solvent used in sample was water, the water molecules absorbed the microwave instantly and rapidly.[46] The drying rate decreased continuously with time and initial moisture content (Fig. 8). During all the experiments (irrespective of power), it was observed that the drying rate was higher for samples with higher initial moisture content, which is impossible to achieve by conventional drying methods. No constant-rate drying period was noticed and the complete drying operation occurred in the falling-rate period, in which the

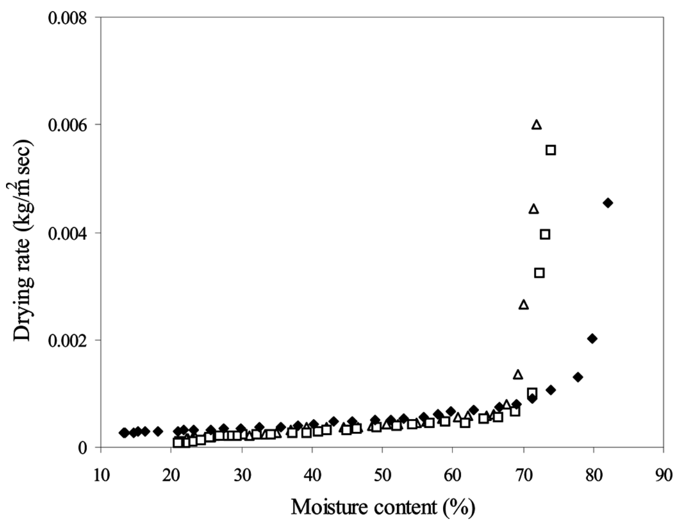


FIG. 8. Drying time versus moisture content of POP at uniform power ranges (180 W): ◆ IM 80%; □ IM 75%; Δ IM 70%.

TABLE 5
Analysis of variance for quadratic model (Eq. (10))

Source	DF	Seq. SS	Adj. SS	Adj. MS	F	P
Regression	9	0.3944	0.3944	0.04382	458.62	<0.0005
Linear	3	0.2471	0.0862	0.02873	300.69	<0.0005
Square	3	0.1335	0.1335	0.04448	465.60	<0.0005
Interaction	3	0.0138	0.0138	0.00461	48.25	<0.0005
Residual error	10	0.0009	0.0009	0.00009	—	—
Lack of fit	5	0.0008	0.00086	0.00017	9.02	0.015
Pure error	5	0.0001	0.000095	0.00002	—	—
Total	19	0.3953	—	—	—	—
S = 0.009775			$R^2 = 99.8\%$		R^2 (adj) = 99.5%	

internal liquid diffusion controls the drying characteristics. Similar results were reported by Panchariya,[47] Maskan,[48] and Wang[49] for the drying of black tea, banana, and apple pomace, respectively.

Optimization through RSM

Central composite design (CCD) was used to develop correlation between the drying process variables (initial moisture content, microwave input power, and drying time) and the percentage moisture removal. A quadratic model was chosen to represent the present data on percentage removal of water in plaster. An empirical relationship between the response and independent drying variables was developed by multiple regression analysis using the response obtained for the design matrix developed through CCRD, which was expressed in the following quadratic form:

$$\begin{aligned} Y_1 = {} & 1.33945 - 0.01295A + 4.48 \times 10^{-5}B \\ & - 5.54 \times 10^{-3}C + 9 \times 10^{-5}A^2 - 1 \times 10^{-5}B^2 \\ & + 1 \times 10^{-5}C^2 + 1 \times 10^{-5}AB - 2 \times 10^{-5}AC \\ & + 1 \times 10^{-6}BC \end{aligned} \quad (10)$$

In Eq. (10), a positive sign indicates a synergistic effect, whereas a negative sign designates an antagonistic effect. The quality of the model developed was evaluated based on the correlation coefficient value. Various R^2 values obtained for Eq. (10) are given in Table 5. The values of R^2 that are close to unity have advocated a strong correlation between the observed values and the predicted values. This means that a regression model provides an excellent explanation of the relationship between the independent variables (factors) and the response (% moisture removal). The smaller the standard deviation, the better the model will be, as it will give predicted values that are closer to the actual value for the response.[50]

The adequacy of the models was further justified through analysis of variance (ANOVA). The ANOVA for the quadratic model for drying rates is listed in Table 5, from which the $F_{statistics}$ values for linear, squared, and interaction terms were 300.69, 465.60, and 48.25, respectively. For the overall regression, the $F_{statistics}$ value was 458.62. These large values imply that the drying process of plaster can be adequately explained by the model equation.[51,52] From Table 6 it was observed that the

TABLE 6
Analysis of variance (ANOVA) for the individual terms present in the response surface quadratic model (Eq. (10)) for percentage moisture removal

Term	Coeff.	SE Coeff.	T	P
Constant	1.33945	0.140357	9.543	<0.0005
A	−0.01295	0.004004	−3.234	0.009
B	4.48×10^{-5}	0.000170	26.342	< 0.0005
C	-5.54×10^{-3}	0.000292	−19.012	< 0.0005
A^2	9×10^{-5}	0.000026	3.554	0.005
B^2	-1×10^{-5}	0.000000	−35.574	< 0.0005
C^2	1×10^{-5}	0.000000	22.513	< 0.0005
AB	1×10^{-5}	0.000001	4.040	0.002
AC	-2×10^{-5}	0.000002	−9.428	< 0.0005
BC	1×10^{-6}	0.000000	6.288	< 0.0005

values of P were less than 0.005, which indicates that the individual model terms present in the model were significant. In this case, the process variables initial moisture content (A), microwave power input (B), and drying time (C), the interaction terms AB, AC, BC, and the square terms A^2, B^2, C^2 are more significant to the response.

Interaction Effects of Operating Variables

The moisture removal for plaster by microwave drying at different degrees of the variables were plotted in three-dimensional response surfaces and contour plots (Figs. 9–11), which corresponds to a myriad number of combinations of the two selected variables, while the other factors remain at their zero level. All the response surface plots reveal that at low and high levels of the variables the moisture removal rate was minimal; however, it was noted that a region existed where neither an increasing nor a decreasing trend in the drying rate. The nature of

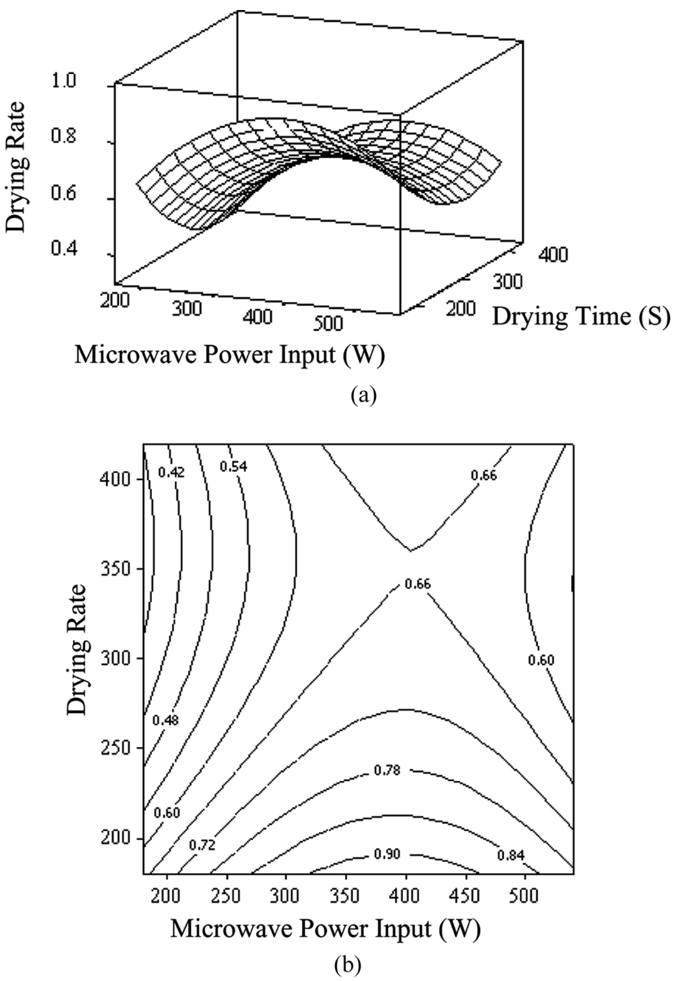


FIG. 9. Interactive effects of varied drying time and microwave power input at initial moisture content of 75% drying rate of POP. (a) 3D surface plot; (b) contour plot.

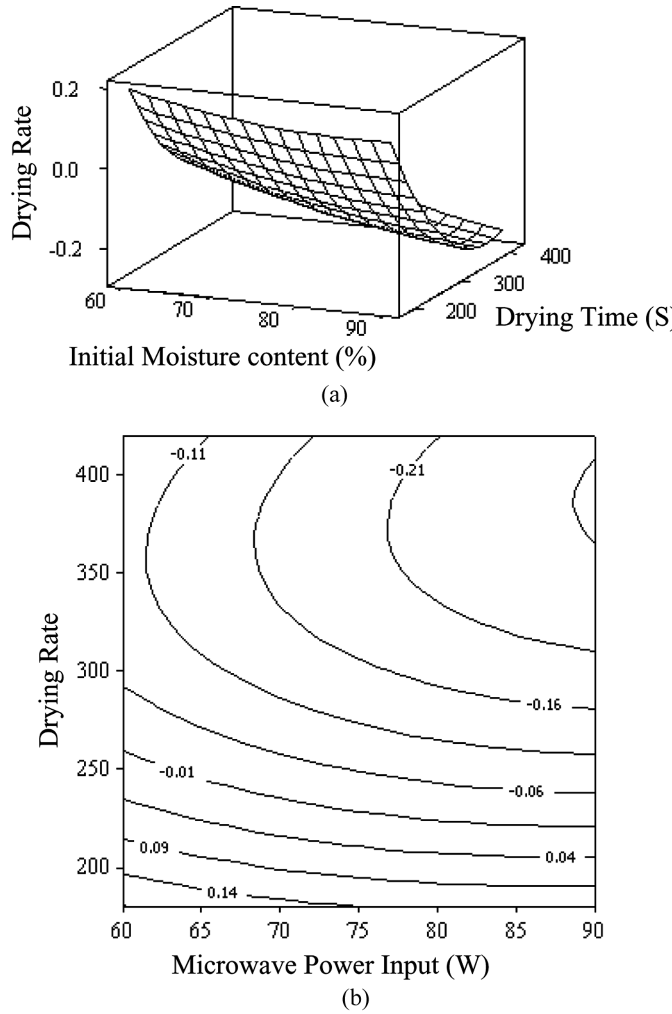


FIG. 10. Interactive effects of varied drying time and initial moisture content at microwave power input of 360 W on drying rate of POP. (a) 3D surface plot; (b) contour plot.

all plots shows the existing mutual interaction between the selected variables. Based on the F and P values (Tables 5 and 6), all the variables were found to have significant interaction effects on moisture removal.

Contour curves represent the drying rate as a function of moisture contents of two independent variables with another variable being at a fixed level. Figures 9a and 9b show the combined effect of microwave input power and drying time on the drying of plaster at constant initial moisture content of 75%. It was observed that the decrease in microwave input power and increase in drying time increases the drying process of plaster, according to the polynomial equation (Eq. (10)) represented by the term BC. The saddle contours indicate that the drying rate was decreased at the optimal region, the lowest drying rate was generally obtained at the intersection of the optimal values. Under strong microwave power conditions (e.g.,

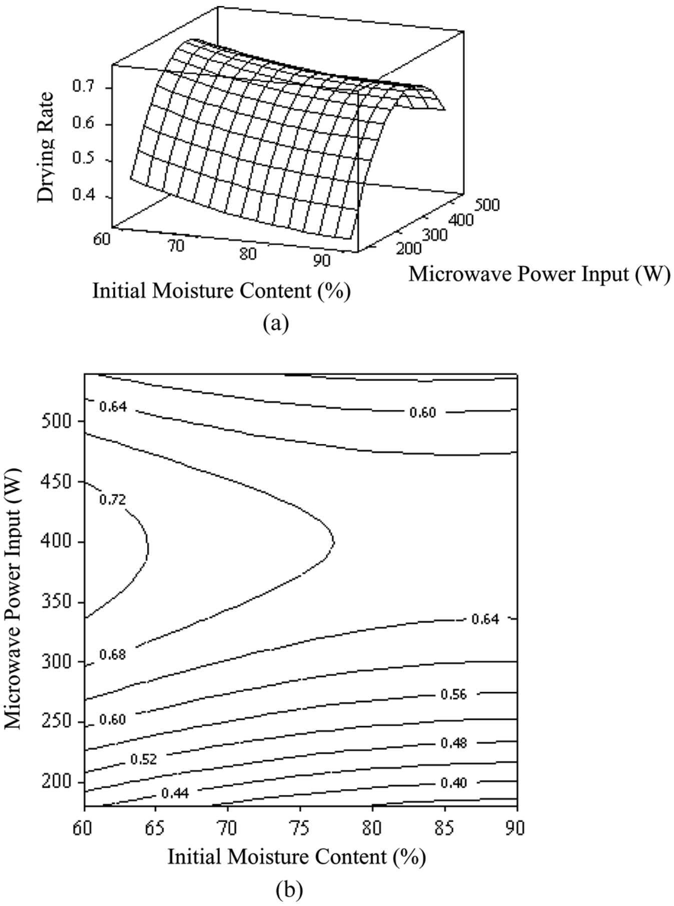


FIG. 11. Interactive effects of varied microwave power onput and initial moisture content at drying time of 300 s on drying rate of POP. (a) 3D surface plot; (b) contour plot.

microwave input power >180), the calcium ions were mostly in the form of $CaSO_4^+$, which was highly effective for decreasing the drying rate, pressure effect, and hydration effect.[53] The tortuous surface in Figs. 10a and 10b showed the interaction effect of drying rate on initial moisture content and drying time, indicating that high initial moisture content and greater drying time led to higher drying rates when the microwave power input was maintained at 180 W. The same phenomenon was found when microwave was applied for pectin extraction from apple pomace.[54] A relatively strong interaction between drying rate on initial moisture content, which was reflected by the corresponding p value (0.0005) deduced from elliptical nature of the contour curve and showed an increase in drying rate. On the other hand, the effect of variation of initial moisture content and the microwave input power on moisture removal is shown in Figs. 11a and 11b. When the drying time was set at 300 s as the center point showed an increasing trend for high initial moisture content of plaster and less microwave power input, respectively. The curved contour shows the existing interaction between initial moisture content of sample and microwave input power.

The stationary point or central point is the point at which the slope of the contour will be zero in all directions. The coordinates of the central point within the highest contour levels in each of these (Figs. 9b–11b) correspond to the optimum values of the respective constituents.[53] Thus, the optimization was made for the maximum moisture removal ratio of plaster for the independent operating variables as follows: microwave input power (B) 180 W; drying time (C) 480 s; initial moisture content (A) 60%. Further, an experiment was conducted at the predicted optimum conditions to validate the model. The experimental moisture removal was 89.2%, which was close to the computed value of 90.0% using Eq. (10). From the response surface analysis, it was confirmed that the RSM could be effectively used to optimize the process variables for the microwave drying process using the statistical design of experiments.

CONCLUSION

The drying characteristics of plaster of Paris (POP) were made under microwave conditions using three different levels of power input, initial moisture at various exposure time for rectangular-faced cuboids. The measured moisture ratio was analyzed for its dependency on the above-mentioned variables. The change in the surface structure during microwave drying was also analyzed with the help of XRD and SEM photographs. The kinetic studies were also carried out at different microwave power input levels. The effective diffusivity of the moisture during the microwave drying process was also estimated for different operating conditions. A model was proposed to represent a microwave drying process of plaster of Paris using RSM and the results of the statistical data were reported. The optimization of the drying process was made using RSM. The optimum values of the independent variables for the maximum moisture removal ratio of plaster of Paris are initial moisture content (A) 60%, microwave input power (B) 180 W, and drying time (C) 480 s. The present study should be useful for the design and scale-up of the microwave drying process of nonconductive as well as porous materials in particular for ceramic industries.

NOMENCLATURE

A, B, C	Independent variable
B	Breadth (mm)
D_{eff}	Effective moisture diffusivity ($m^2 \cdot s^{-1}$)
E	Electric field strength (V/m)
f	Microwave frequency (2450 MHz)
H	Height (mm)
k	Constant in Eqs. (1) and (2)
L	Length (mm)
M_e	Equilibrium moisture content (g/g (dry basis))

M_0	Initial moisture content (g/g (dry basis))
M_t	Moisture content at specific time (g/g (dry basis))
N	Total number of experiments
n	Number of factors
P_V	Power density (W/m^3)
R^2	Coefficient of determination
T	Half of thickness of plaster of Paris (m)
t	Drying time (s)
W	Power (W)
$x_i x_j$	Independent variables
Y	Dependent variable
Y_i	Predicted response

Greek terms

β_i	*i*th Linear variable coefficient
β_{ij}	*ij*th Interaction coefficient
β_0	Regression coefficient at center point
β_1,β_2,β_3	Linear coefficients
δ	Dissipation factor
ε'	Dielectric constant
ε''	Dielectric loss

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