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Dehydration characteristics and drying quality of Plaster of Paris using microwave heating process.

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ABSTRACT : Conventional heating or drying involves exposure of materials to high temperature and for long times, which can result in serious damage to texture, color and other properties of the treated material. In recent years, microwave drying has gained popularity as an alternative drying method for a wide variety of products. The drying system was operated in the microwave power range of 180 – 540 W, having plaster of paris slices of 13-18 mm thickness, with 70 to 90% initial moisture content. The drying characteristics of Plaster of paris, under varied microwave condition were studied for different shapes of materials through various drying parameters like microwave power, initial moisture content and drying time. The overall picture of dehydration characteristics, the mechanism of their hydration and the crystal growth, the role of the size and stability of critical nuclei, the different crystal faces and step edges of Plaster of paris were discussed.

Keywords : Microwave drying, Dehydration, Plaster of paris, Texture.

Introduction : Plaster of Paris, a basic salt of calcium sulfate with half molecule of water of crystallization ($CaSO_4$ ½ H_2O) is made by calcining the mineral gypsum. When the powdered hemihydrate is mixed with water to form a paste or slurry (also called a slip), the calcining reaction is reversed and a solid mass of interlocking gypsum crystals with moderate strength is formed. Upon setting there is very little (a slight contraction) dimensional change, making the material suitable for accurate molds. The hardened mass is not a compact solid, but a highly porous material with a relatively large internal surface consisting of interlocking crystals in the form of plates and needles. Plaster, known for its porosity and strength, is used as fire resistance on residential and other structures, bone repair cement, ceramic industry for the preparation of models, moulds, gypsum plaster boards and decorative picture frames, interior decoration of buildings and other establishments, etc. The conditions of moisture removal in molded plaster of paris can be changed to adjust the porosity of the hemihydrate, resulting in the formation of alpha and beta hemihydrates, which are chemically identical. The morphology of plaster of paris crystals depend on the formation conditions and the drying methodology put into practice.

Advances in the drying technology of porous material have been increasingly stimulated over the past few years in contrast to the traditional convective drying with warm air (solar drying, open air sun drying, single layer solar drying, convective type tray dryer, etc.), and are becoming more important. The use of microwaves as a source of energy is rapidly growing and getting convenient advantage. Therefore, microwave heating has shown advantages over the conventional heating method in terms of energy efficiency, higher reaction rates and shorten in reaction times [1]. Industrial and geochemical interest has resulted in a considerable amount of research on the mechanism of POP crystal growth, both in the presence/ absence of different types of chemical additives [2]. Further the overall picture of dehydration characteristics, the mechanism of their hydration and the crystal growth, the role of the size and stability of critical nuclei, the different crystal faces and step edges of POP are considered in the literature [3]**.** Few studies deal with the possibility of predicting the rate of heating up the material exposed to microwaves, while others are concerned with the specific acceleration of drying by means of microwaves. Due to the limited amount of experimental work on microwave drying of POP, reported to date, it is therefore, the objective of this study, to investigate microwave drying of POP and create new suitable models including combine effect of drying time and input power.

Materials and methods :

Sample preparation : The commercially available high purity, analar grade POP, less than 50 microns were obtained from SRL, India. POP was then made into paste with carrier water and molded to three different basic geometries, including square (70 x 70 x 15 – L x B x H in mm), rectangle (80 x 70 x 13 – L x B x H **-** in mm) and cylinder (64 x 18 – D x H **-** in mm) approximately weighing 150grams. The freshly molded samples were initially tempered; which allows time for the internal moisture and temperature gradient during the resting period [4], Tempering is done for a period of 18 to 24 hours, so that the material becomes rigid and stable. The experiments were conducted with an initial moisture content of 70-80%, before exposing to microwave drying.

Microwave drying : The microwave-drying oven employed for experimentation was a SAMSUNG C-103F model, with inbuilt bio-ceramic cavity. The microwave generator (352 x 220 x 300-W x H x D - in mm) at 2450 MHz and 900Watts of nominal power has been equipped with a special device allowing continuous supply of microwave power at controllable level from 100 to 900Watt with increments. As the commercially available microwave oven has its own limitation in that of monitoring the sample weight, an inbuilt weighing system was used [5]. Initially the turnable was dismantled and removed from the cavity. A circular opening with a diameter of 25mm was drilled at the bottom of the oven cavity, through which, a teflon made weighing beam was introduced to hold the sample at its top. The bottom of the beam was placed in the load cell (sensitivity ±0.01

grams) so as to measure the load placed in the top of the weighing beam, which is connected to the digital converter through a weighing recorder and a voltmeter. The electronic load cell remained unaffected by the microwave, since they are calibrated priorly. The oven is provided with a magnetron for producing dielectric waves, a fluorescent bulb and a circulating fan for the distribution of the produced microwave. The amount of evaporated water during drying was determined directly from digital screen of drying equipment at about 10 sec intervals until the final moisture content of the sample was attained. The digital converter is connected to the workstation, for the conversion of digital data in to graphical format. The software was developed and programmed to the current requirements (V.I. Microsystems Chennai, India). Proper microwave leak proof agents are provided at the bottom of the cavity where the hole is drilled (to arrest the interaction of microwaves outside the cavity). In addition to that a metallic screen provided around the system, a teflon frame covers the device to avoid further leakage of microwave.

Experimental procedure :

Drying of POP was started with initial moisture content of approximately 70 to 80 % (wet basis), and continued for a period of time with constant power input. All experiments were conducted at three different initial moisture contents (70%, 75% and 80%), power ranges (180W, 360W and 540W), and geometry (square, rectangle and cylinder). The microwave-dried samples were then placed in a hot air oven set at 105ºC until bone dry, finally the weight of the sample was noted. The mass of the samples chosen for the runs was approximately 150 g. The lists of experiments conducted were given in **Table 1**. All experiments were performed in triplicate, the average values of these replications were used for further analysis of microwave drying parameters like drying rate, moisture content at various drying time.

Absorption characteristics of plaster and water

Polar substances absorb microwaves to convert microwave energy to thermal energy, for which the rate of absorption is determined by its dipole rotation and ionic conductance characteristics. The magnitude of thermal energy deposited into a material is dependent on the internal electric field strength, the frequency of the microwave radiation and on the dielectric properties of the material.[6] The primitive equation to calculate the power absorption density per unit volume for dipolar rotation can be approximated, if the electric field strength is known, and expressed as given in Eq. (1) and (2)

$$P_V = kE^2 f \xi' \tan\delta \qquad (1)$$

$$P_V = kE^2 f \xi'' \qquad (2)$$

Where, P_V the power density (W/m^3), k the thermal conductivity, E the electric field strength (V/m), *f* the microwave frequency (2450 MHz), tan δ 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) the dielectric constant (a measure of the charge retention capacity of a medium) and dielectric loss (the imaginary part of the dielectric constant, and determines the lossiness of the medium) respectively. If a material does not have significant magnetic properties, only complex permittivity can be measured and defined by the following equations :

$$\xi^* = \xi' - j\xi'' \quad (3)$$

$$\tan\delta = \left[\frac{\xi''}{\xi'}\right] \quad (4)$$

Where, ε* the complex permittivity (the ability of material to couple electrical energy from a microwave field), $j = (-1)^{1/2}$ and tan δ, the loss tangent. To enlighten the influence of microwave heating, the specific heat, thermal conductivity and dielectric properties of materials were considered (Table 2). At a fixed microwave frequency, only the material properties ε* and tan δ determine the total power dissipated within the sample[32]. Both these parameters depend strongly on temperature. To study the interaction characteristics of plaster with water, raw plaster was spread on the Teflon line sheet, placed into microwave oven and then irradiated at three power levels, The experimental time versus temperature curves for raw plaster, water and molded plaster were plotted for increasing power inputs in microwave system.

Results and discussion :

Characterization : The internal heat generation influences heat and mass transfer effects on samples with a wide change in external as well as internal characteristics of POP, which leads to the estimation of few physical properties including porosity, bulk density, linear shrinkage (square, rectangle), radial shrinkage (cylinder), and volumetric shrinkage for wet and dried samples. A brief note on the basic characteristics of the sample before and after microwave drying was revealed in T**able 3.** When POP was subjected to microwave atmosphere, the porosity of plaster of paris increased after exposure to microwave drying. An average increase of 5% in porosity was noted for all samples, irrespective of geometry, initial moisture content and power. The porosity of fresh tempered samples varied from 41.20 to 71.0%, where the dried samples were in a range of 43.0 to 76.0%. Bulk density, an important physical property in characterizing the texture and the quality of dry and intermediate moisture materials is essential in modeling and design of various heat and mass transfer operations. The bulk density (kg/m^3), decreased in dried samples while compared to the fresh ones. The bulk density of POP reduced from a minimum of 0.997 to 0.830((kg/m^3) and from a maximum of 2.11 to 1.138(kg/m^3). No significant change in color was observed between the fresh and dried samples. The dried samples were much brighter than moist samples.

Microstructure determination : The internal heat generation influences the heat and mass transfer effects on samples in external as well as internal characteristics of plaster. Porosity has a well-defined effect on the physical properties, such as mass diffusion coefficient, thermal conductivity, and thermal diffusivity during drying process. The material when subjected to microwave irradiation, losses some of the volatile matter and moisture content. The dried plaster of paris contains numerous pores within it, forming an open structure [8,9]. Microwave causes violent evaporation of water in pores followed by a collapse of porous structure and partial disconnection of pores. SEM micrographs (Fig 1) showed that the sample was almost fully dense with large grains in the central part. Porosity is strongly affected by the drying method and conditions, an increase in porosity was observed for the materials dried. Microwave irradiation is a process by which, the inherent moisture content of any material can be reduced, the movement of the water molecules is from the centre of the material to the outer surface, and this creates an increased porous microstructure in the irradiated material.

Adsorption characteristics :

Plaster is comparatively a poor microwave absorber, since the carrier (water) used to bind, is an excellent absorber, a quick warm up of the sample has occurred. When raw water was placed in microwave oven, the temperature of water molecules rose rapidly and boiling point was attained in 300 sec at minimal power (180W), while the same conditions reached before 180 sec at 540W(Fig 2). When the temperature of raw plaster increased gradually from room temperature to a maximum of 42°C at 300 sec by 180 W, a maximum temperature of 35°C was obtained by 540 W power (Fig 3). A few minutes exposure of the molded plaster, even at low power levels raises the temperature of the slab above 50°C for all power ranges. The molded plaster samples processed for 300 sec at 180, 360 and 540 W., showed a significant raise in the temperature. Thus a maximum temperature of 76°C was achieved at 180W (Fig 4). This strategy may be advantageous for achieving a greater heating rate because the water molecules that convert microwave energy to heat, when it starts to evaporate, as a result of this heat and the material begins to dry.

In microwave heating, regions of higher moisture content absorb more microwave energy, where heat is generated throughout the material[10]. According to this study drying of plaster begins approximately 20 s after the operation commences, a steady-state conversion is attained. As seen from Fig. 4 in Microwave heating complete drying of plaster was achieved within only 20 sec at 180W. Moreover, microwave heating provides great time savings, compared with conventional drying. This observation was confirmed by the analysis of the XRD patterns of the samples. X-ray diffraction finds the geometry or shape of a molecule using x-rays and is based on the elastic scattering of x-rays from structures that have long range order. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction [11]. While powder X-ray diffraction of sample essentially showed peaks corresponding to Calcium sulphate in Fig. 5(a), whereas in Fig. 5(b) the powder X-ray diffraction of microwave-irradiated product essentially showed peaks corresponding to calcium sulphate with large number of peaks and more d-spacings at 2 theta, mentioning the scattered pattern during the interaction of water molecules with crystals of calcium sulphate.

Conclusion :

The microwave drying of plaster of paris showed certain parameter changes during its operation. Based on the above analysis the following conclusions were drawn. A total drying time reduction is possible if microwave techniques are improved. The microwave drying of the plaster of paris was performed at different conditions and the moisture ratio obtained through the experiments was fitted to the various thin-layer models available in the literature. While drying samples in oven, the surface alone is exposed to larger driving force for mass transfer, than particles in the base of the samples. Since the heating source is by microwave this phenomena is vanished due to the basic property of microwave that it dries the particles right from the center, which is an added advantage of microwave drying. In addition to their ability to reduce drying time microwave oven offers several other advantages over thermal ovens, unit cost is less than that of conventional ovens, running cost are low, there is a modest saving in bench space an a minimal effect on room temperature.

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Table (1) : Summary of experiments for drying plaster of paris.

Expt No.	Microwave power (W)	Initial Moisture content (%)	Sample Geometry	Surface area (m^2)	Depth (m)
1 2 3	180 360 540	70	Square	0.0091	0.015
4 5 6	180 360 540	75			
7 8 9	180 360 540	80			
10 11 12	180 360 540	70	Rectangle	0.0099	0.013
13 14 15	180 360 540	75			
16 17 18	180 360 540	80			
19 20 21	180 360 540	70	Cylinder	0.00514	0.018
22 23 24	180 360 540	75			
25 26 27	180 360 540	80			

Table (2) : The thermal and dielectric properties of plaster and water, as collected from (Abdullah Seryrankaya).

Sl.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	-

Table (3) : Characteristics of Plaster sample before and after microwave drying.

Sl.No.	Power (watt)	Moisture content (%)		Porosity (%)		Bulk Density (gm/cc)	
		Initial	Final	Initial	Final	Initial	Final
Square (70 x 70 x 15 – L x B x H - in mm)							
1	180	69.16	27.92	58.41	61.15	1.49	1.04
2	360	70.04	33.06	63.12	66.56	1.19	0.94
3	540	69.92	43.06	62.19	65.00	1.64	0.92
4	180	75.42	25.16	71.00	76.21	1.92	1.23
5	360	74.63	31.43	41.31	43.00	1.33	0.91
6	540	75.41	38.73	53.12	56.62	1.33	0.90
7	180	80.90	17.22	57.76	65.72	2.11	1.14
8	360	80.42	28.62	58.74	62.52	1.20	0.94
9	540	80.15	29.36	60.21	63.06	1.82	1.12
Rectangle (80 x 70 x 13 – L x B x H - in mm)							
10	180	70.06	22.16	60.06	62.23	1.29	0.79
11	360	70.15	28.55	51.02	56.16	1.20	0.96
12	540	70.63	39.93	56.93	61.19	1.54	0.77
13	180	74.11	20.97	56.31	63.22	1.49	1.04
14	360	75.00	27.41	52.14	61.36	1.51	0.87
15	540	74.42	32.93	41.20	49.35	0.99	0.83
16	180	80.17	13.23	52.12	61.71	1.12	0.88
17	360	79.43	23.00	57.47	61.06	1.80	0.97
18	540	81.83	27.86	61.42	69.43	1.02	0.82
Cylinder (64 x 18 – D x H - in mm)							
19	180	69.26	32.40	42.49	49.06	1.05	0.79
20	360	79.49	39.13	54.04	59.19	1.15	0.85
21	540	70.17	52.17	51.18	56.13	2.00	1.57
22	180	75.25	26.77	59.30	62.06	1.09	0.84
23	360	74.92	37.62	62.03	68.14	2.02	1.68
24	540	75.07	44.73	41.63	45.02	1.05	0.97
25	180	79.17	21.68	48.02	55.00	1.16	0.84
26	360	79.47	34.52	46.41	50.47	1.05	0.99
27	540	80.52	41.94	42.06	45.15	1.42	0.89

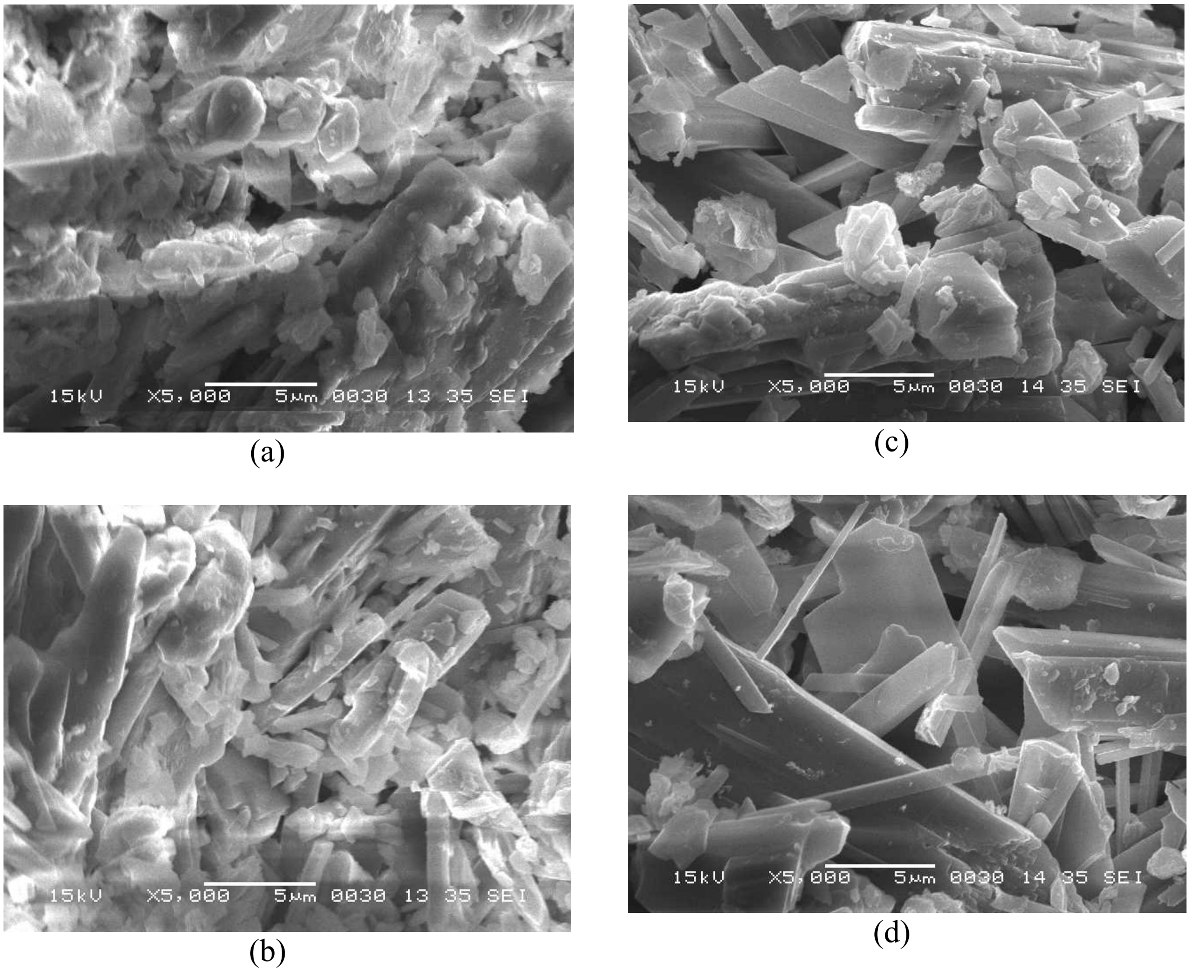

Figure (1) : Scanning electron micrographs of dried plaster cuboids :
(a) 14mm. (b) 13mm. (c) 12mm. and (d) 11mm.

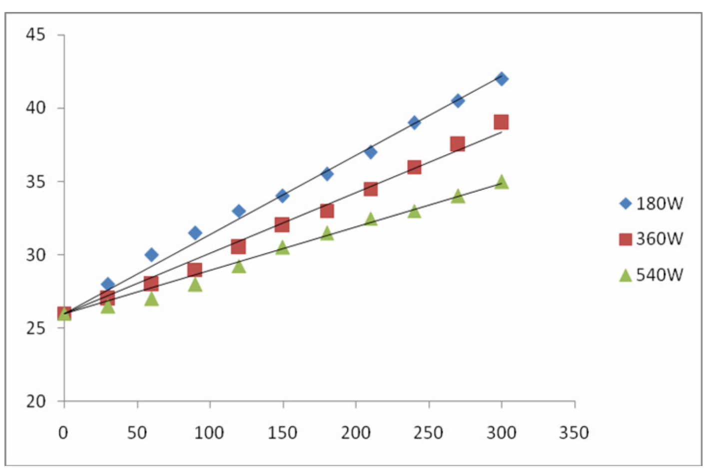

Figure (2) : Temp (C) Vs Time (S). Effect of power dependent microwave heating of plaster : ♦ 180W: ■ 360W: ▲ 540W

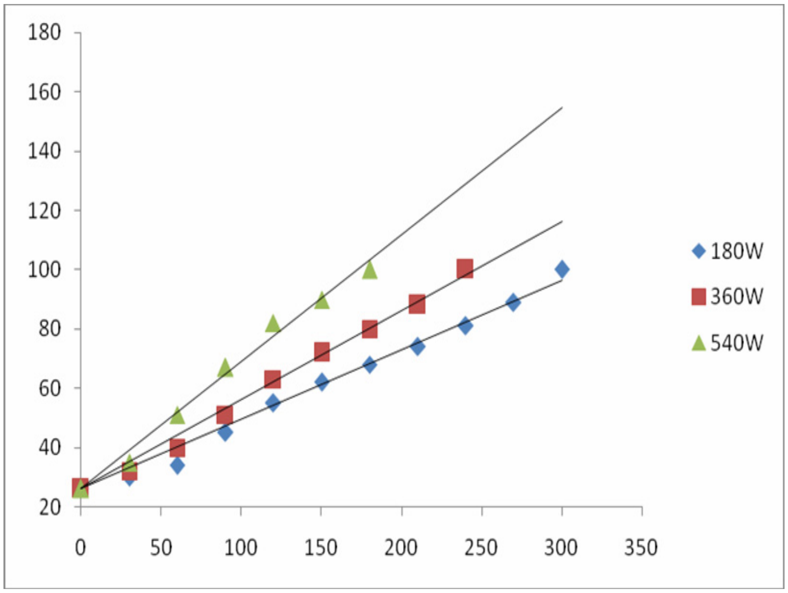

Figure (3) : Temp (C) Vs Time (S). Effect of power dependent microwave heating of water : ♦ 180W: ■ 360W: ▲ 540W

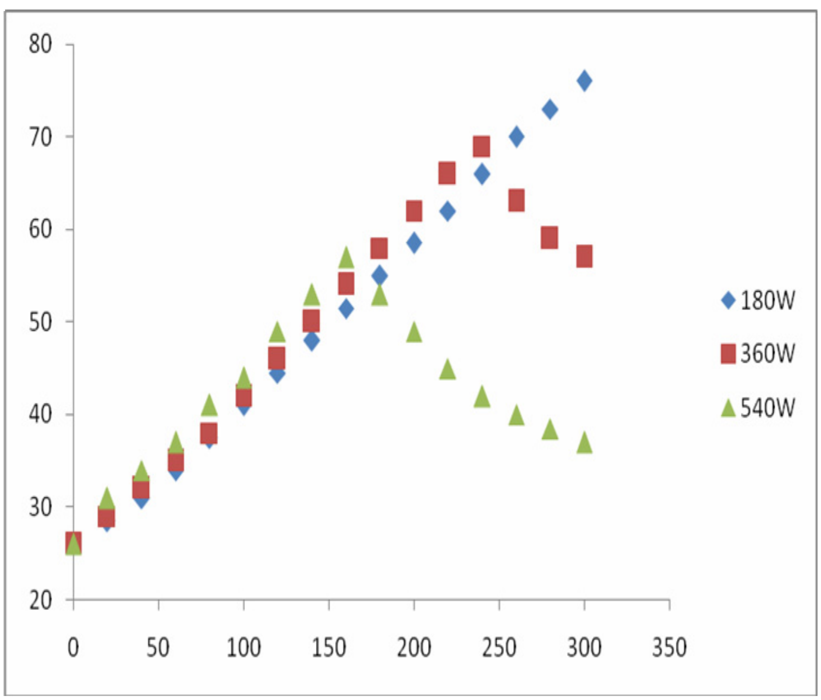

Figure (4) : Temp(C) Vs Time (S). Effect of power dependent microwave heating of water on plaster : ♦ 180W: ■ 360W: ▲ 540W

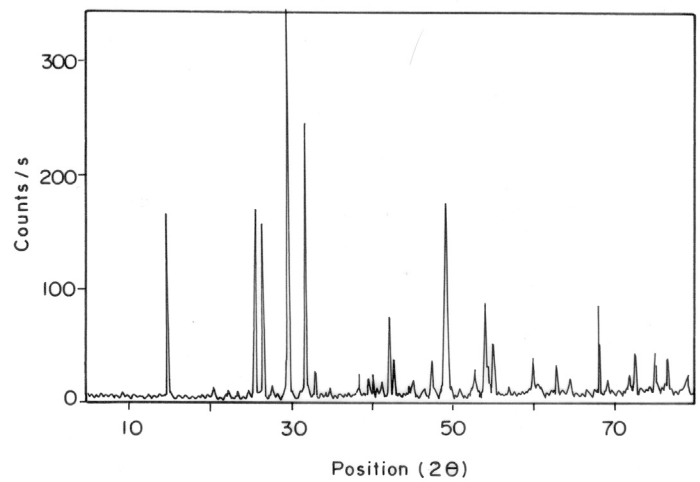

Figure (5a) : XRD pattern of plaster before Microwave irradiation.

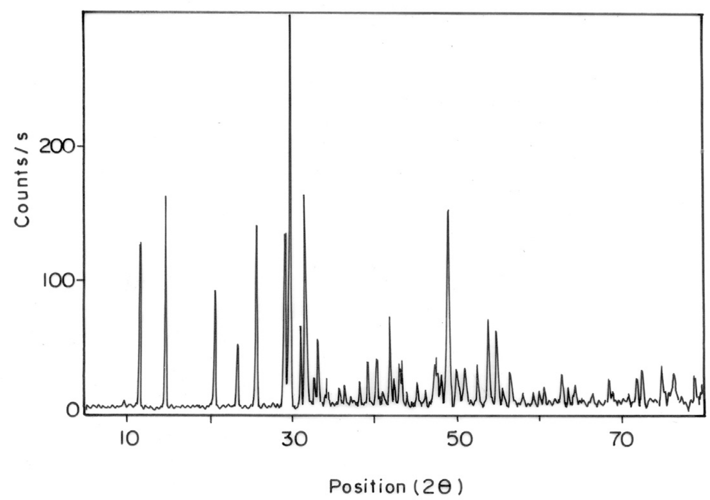

Figure (5b) : XRD pattern of plaster after Microwave irradiation.