Int. J. Appl. Ceram. Technol., 5 [1] 94–100 (2008)

International Journal of Applied Ceramic Technology
Ceramic Product Development and Commercialization

A Comparison of the Effects of Microwave Versus Conventional Drying on the Mechanical Properties Distribution of Dried Green Porcelains

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The effect of fast microwave drying of electroporcelain insulator component was studied by determining the reliability parameter. The Weibull modulus was calculated using the three-point bend strength data of a large number of green samples which were dried using microwave energy. The results were compared with those obtained by conventional drying methods. It was observed that in most cases, microwave-dried components yielded higher Weibull modulus than their conventionally dried counterparts. A high modulus value of >15 was achieved on the microwave-dried samples. The analysis of the result was useful in understanding the fast drying process in ceramics.

Introduction

The conventional drying of large ceramic components is a time-consuming process and involves a combination of natural drying and tunnel drying. Typically, for a porcelain insulator of >15 kg of weight, a natural drying of 5–7 days followed by tunnel drying of 5–7 days are required to remove 15–18% moisture. Very fast drying by conventional methods is not possible due to the defect generation during fast removal of water from the wet core through the dried surface. Porcelain manufacturing is a traditional process utilizing a mixture of natural raw materials like clay and quartz and synthetic raw materials like alumina. The conventional drying process has many limitations including the high drying time, high energy cost, large space requirement, low turnover, and high manpower requirement. All these factors lead to high cost of the product which is not feasible to survive in today's competitive market. In addition, there is a threat to these industries due to the environmental pollution arising from the burning of fossil fuels during processing. Therefore, a need arises to develop a fast, efficient, and environment friendly process, which suitably replace the conventional technology of drying in a commercial scale. Microwave drying has proven to be such a technology for the future growth of these industries.

Though the microwave drying on ceramics has been attempted by many researchers and industries around the globe,[1–10] the commercialization of this process in ceramic industry for fast drying large and complicated articles has not yet succeeded. One of the

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Table I. Typical Electroporcelain Compositions Studied in this Work

Composition	A	B	C	D
Feldspar; $d_{50} \sim 70\,\mu m$	18–22	10–15	22–30	5–10
Clay (a mixture of two or three different clays)	45–55	45–55	44–50	35–45
Quartz; $d_{50} \sim 150\,\mu m$ and $\sim 50\,\mu m$ (a mixture of both)	25–30	20–25	—	10–15
Alumina; $d_{50} \leq 5\,\mu m$	—	—	25–35	20–28
*Others (pyrophyllite, felcite, sericite, etc.)	2–5	12–18	—	4–8

All the raw materials were sourced from different parts of India. *Minor raw materials.

main reasons for the same besides the cost aspect is that the effect of microwave drying on the mechanical properties of these materials has not been studied in the green state. Further, there are conflicting data in the literature describing the microwave process as detrimental in drying ceramic components.[7]

In the present study, the three-point bend strength data of a large number of samples for four different porcelain insulator compositions have been studied and the scatter in data is analyzed statistically to obtain the Reliability parameter the Weibull modulus. The porcelain insulator was chosen in this study because; the insulators do not tolerate any defects due to their use in very critical high voltage applications.

Experimental Procedure

Commercially available proprietary electroporcelain insulator compositions were used in this study. Two quartz-based and two alumina-based compositions were chosen. The typical compositions are given in Table I. The raw materials were ball milled in wet condition using river pebbles. The slurry was filter pressed and the cake was used for extruding bars of 150 mm length and 12 mm diameter using a vacuum extruder. The green bars after extrusion were dried in air for 1 h to impart sufficient strength to handle. These bars were used for drying in an electrical oven by conventional drying method at a peak temperature of 120°C for 2 h with a total cycle time of 8 h. Similarly, a set of different samples from the same batch were simultaneously microwave dried at a maximum surface temperature of 70°C with a total cycle time of 4 h. The bar samples were hanged from a porous substrate and kept in an isothermal barrier casket (Fig. 1) inside a 6 kW microwave furnace of M/s Cober Inc. (Norwalk, CT) make. A constant microwave power of ~ 0.5 kW was used for all the experiments. The initial moisture was $\sim 16\%$ and the final level was $\leq 0.5\%$ as confirmed by the standard moisture analysis using moisture analyzer (MX 50, M/s A & D Co. Ltd., Tokyo, Japan) with infrared heating system. In this method, 10 g of the powder was spread on the balance pan and heated by infrared heating system. The equipment monitors the moisture loss continuously at 110°C and automatically stops after all the moisture is removed. In a typical experiment, for 10 g sample with a moisture content of 7%, the time required is 6 min.

In both the conventional and microwave drying cases, the experiments were carried out using airless drying method. The temperature measurement was carried out using a K-type thermocouple after the microwave power was switched off at different time intervals during the drying process. The maximum surface temperature of ~ 70°C was confirmed after similar experiment was conducted and the online temperature was measured by an Optris, Berlin, Germany make infrared pyrometer. The dried samples were cut into a dimension of 120 mm in length for strength measurement. The three-point bend strength of these green dried bars was carried out using a Lloyd Universal testing machine following IEC 60672-2 (1999) standard. The test jig

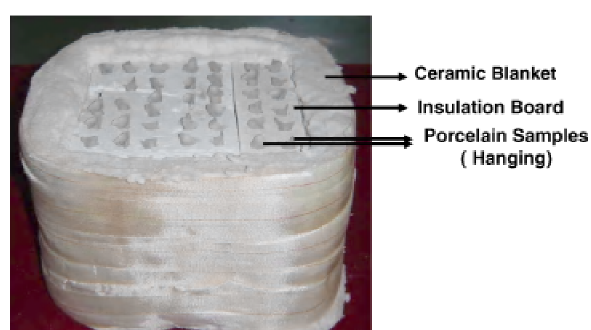


Fig. 1. The casket assembly for positioning the sample for microwave drying experiments.

consists of two parallel test piece support rods upon which the test piece is laid, and one loading rod to apply a force to the test piece, midway between the support rods. The force is applied orthogonally to the plane of the test piece and/or support rods. The span length was 100 mm. The peak force is recorded and then the three-point bend strength is calculated using the formula in equation 1.

$$\sigma_f = (8 \times F \times l)/(\pi \times d^3) \tag{1}$$

where, σ_f is the flexural strength in MPa; F is the total force applied to the test piece at fracture in N; l is the distance between support points in mm; and d is the diameter of round section test piece in mm. At least 30 samples in each batch and in each method were used for comparing the results. The strength data was used for statistical analysis for determining the Weibull modulus in the following way: n number of samples was tested for strength (σ). The values thus obtained were ranked in ascending order $j = 1, 2, 3, 4, \ldots\ldots n$ and lnσ was calculated. The probability of survival P_{sj} for each tested sample was calculated.

$$P_{sj} = (1 - F) = 1 - [j/n + 1)], \text{ where } F \sim j/(n+1) \tag{2}$$

The slope of the line $\ln\{\ln[1/P_{sj}]\}$ vs lnσ was determined which is the Weibull modulus "M." The Weibull modulus "M" is a reliability indicator for a given volume under a given stress.

The modulus of elasticity (MOE) was calculated by measuring the resonant frequency of the green bar samples of 120 mm long and 12 mm diameter using Elastosonic equipment (M/s DEPA Analyzer, Bangalore, India). The tests were carried out following ASTM standards C 1259 (1998) and E1876 (2001) and Young's modulus was calculated using the DEPA software.

Results and Discussions

The cylindrical rods of 12 mm diameter and 150 mm length after extrusion were dried both by conventional electrical heating and by microwave heating to reduce the initial moisture of ~16% to final moisture of ~0.5%. It was noted that, the duration required to dry these items in both the processes are not significantly different; 4 h in microwave compared with 6 h in conventional drying. However, as described elsewhere,[10]

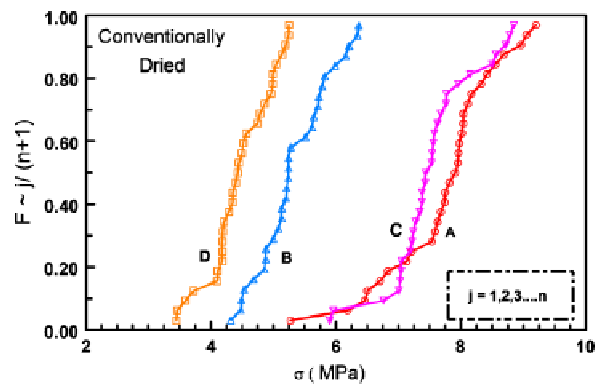


Fig. 2. Weibull curve plotted from experimental data for all the four compositions dried by conventional method.

the conventional drying of large bodies requires >200 h, which can be minimized to ~15–25 h using the microwave furnace. Hence, the effect of microwave is more predominant in the commercial samples with uneven core and shed structure and with high L/D ratio.

The most popular means of characterizing the flaw distribution is by the Weibull approach.[11] It is based on the weakest link theory, which assumes that a given volume of ceramic under a uniform stress will fail at the most severe flaw. The probability of failure as a function of volume can be easily plotted from experimental data by estimating F by $j/(n+1)$, where j is the ranking of the sample and n is the total number of samples. This is plotted against strength values in Figs. 2 and 3 for conventionally dried and microwave-dried samples, respectively, of all the compositions.

The curves in Figs. 2 and 3 provide only an approximation of the probability of failure and do not

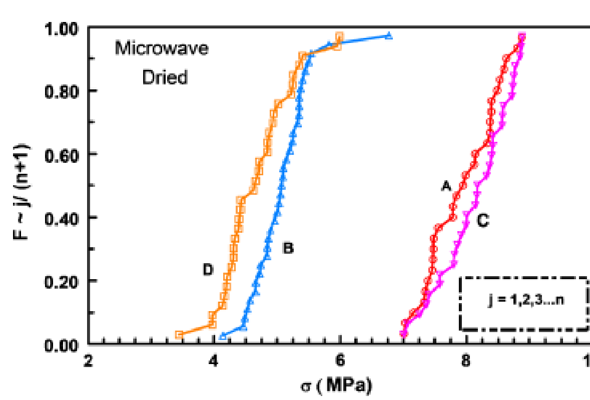


Fig. 3. Weibull curve plotted from experimental data for all the four compositions dried by microwave method.

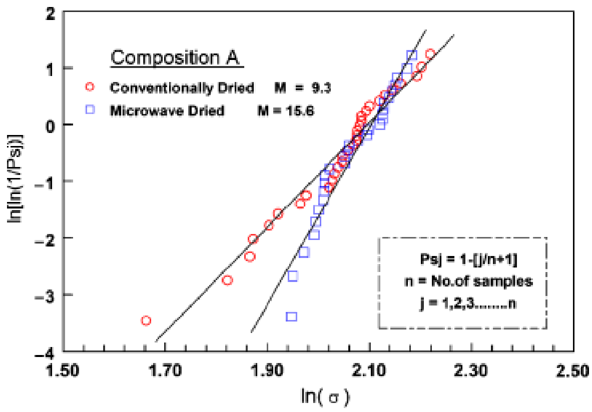


Fig. 4. Weibull plot for composition A (conventionally dried and microwave dried).

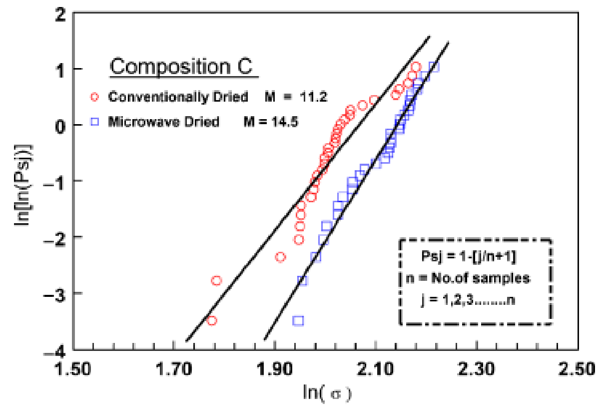


Fig. 6. Weibull plot for composition C (conventionally dried and microwave dried).

yield the *m* value. However, the plots provide a rough estimation of the threshold value for the failure. The threshold stress below which the probability of the failure is zero can be estimated from these plots by extrapolating the curves to *x*-axis. It was observed that the threshold stress of microwave-dried samples of composition A and C are higher compared with their conventional counterparts and almost similar in other two compositions. This analysis implied that microwave-dried samples can have narrow scattering in strength data compared with their conventional-dried counterparts.

The Weibull modulus *M* can be calculated from the slope of the best fit line of the plot of $\ln\{\ln(1/P_{sj})\}$ vs $\ln(\sigma)$. Figs. 4–7 depict such plots of all the four compositions dried by both the methods. The slope of each of the plot as the Weibull modulus was calculated from the best fit data. The results are summarized in Table II. Few observations were made from the plots as given in Figs. 4–7 and the Table II. Firstly, the mean three-point bend strength of all the four compositions dried by microwave method are either comparable or higher compared with their conventional counterparts. This implies that the microwave drying is not detrimental for fast drying of the insulator components. This is the first report of the Weibull modulus in porcelain dried by microwave method. Secondly, the Weibull modulus values for the compositions A, B, and C are substantially higher than their conventional counterparts and those for the composition D was comparable. These results indicate that the microwave drying process is more reliable in drying porcelain components compared with the conventional processing methods. Thirdly,

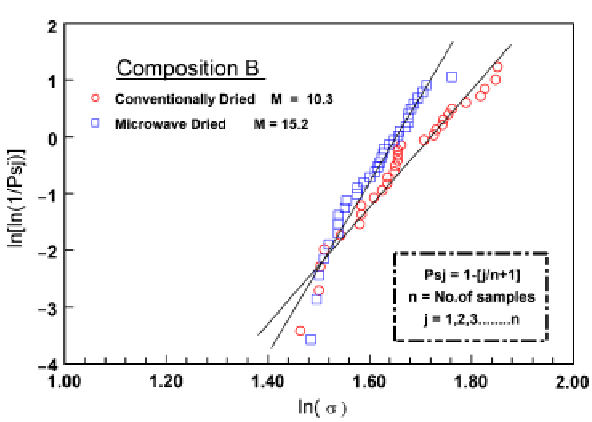


Fig. 5. Weibull plot for composition B (conventionally dried and microwave dried).

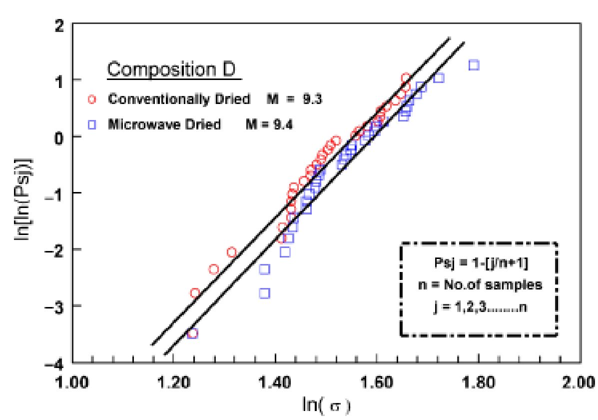


Fig. 7. Weibull plot for composition D (conventionally dried and microwave dried).

Table II. MOR and Weibull Modulus (*M*) for Four Compositions

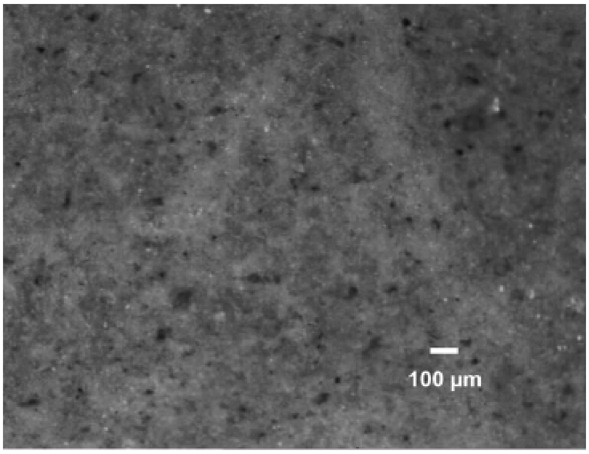
	Composition							
	A		B		C		D	
	Conventional	Microwave	Conventional	Microwave	Conventional	Microwave	Conventional	Microwave
Properties								
Three-point bend strength (MPa)	7.72	7.79	5.35	5.12	7.02	8.0	4.44	4.68
Weibull modulus "*M*"	9.3	15.6	10.3	15.2	11.2	14.5	9.3	9.4
Correlation coefficient "*R*"	0.98	0.97	0.98	0.97	0.96	0.99	0.98	0.97

the weibull plots in the Figures 4–7 suggest that there is monomodal distribution of the defects inside the dried samples. This is confirmed from the co-relation coefficient (*R*) values of the best fit line in calculating the modulus "*M*." As given in Table II; the *R* values lie in the range of 0.96–0.99. This implies that there are no two types of flaws present in these bodies during drying by either method. Fourthly, the very high values of *M* in many compositions indicated that microwave drying of porcelain insulators are not detrimental at a component level and can be used in the industry. The Weibull modulus values for the microwave-dried components were very high keeping in view the traditional nature of porcelain components in mind. Typically, the Weibull Modulus *M* for fired Kaolinites are in the range of 3–13[12] and that for porcelain dental materials in the range of 3–4.[13] A value of 9–16 is very common for advanced ceramics[14] and for metals the typical value of *M* lies above 20.[15] Considering these numbers, the values obtained in this study for clay-based ceramics which were microwave dried are very high and thus implies the better reliability of the process. The microwave drying process is volumetric in nature thus ensuring uniform drying throughout the component resulting in less scatter of data and hence large Weibull modulus. The use of probabilistic design allows a trade-off in material selection between high strength and low scatter. In the present work, microwave drying of porcelain insulator provides low scatter in data.

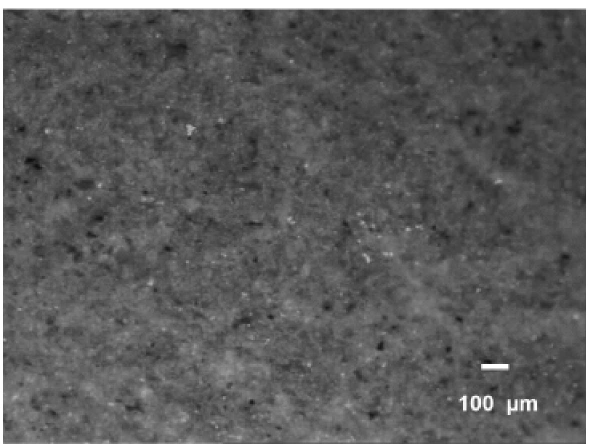
The MOE of all these compositions were also measured and tabulated in Table III. It was observed that similar trend was observed in MOE values as observed for MOR and the Weibull modulus. The MOE values are significantly higher in compositions A, B, and C and comparable in the composition D. This result is another direct evidence of compatibility of microwave drying of porcelain insulator components. The correlation of

Table III. Modulus Elasticity of Different Porcelain Compositions Dried Using Both Conventional Method and Microwave Method

Composition	Conventionally dried (GPa)	Microwave dried (GPa)
A	6.5	7.9
B	4.7	6.2
C	7.0	7.5
D	6.1	6.1



Conventionally dried



Microwave dried

Fig. 8. Optical micrographs of conventionally dried and microwave-dried porcelain sample of composition A.

Young's modulus with strength has already been reported earlier for alumina ceramics.[16] In this work, it has been shown that correlation of strength and Young's modulus behavior exists also with the Weibull modulus in porcelain materials.

The optical micrographs of the bars of composition A dried both by conventional and microwave method revealed equivalent microstructures (Fig. 8). The black spots which represent pores are more uniform and relatively finer in microwave-dried samples compared with the same in conventionally dried counterparts. This is also a positive evidence of uniform drying during microwave processing.

This analysis was further extended to microwave dry commercial insulator components.[10] The drying of the commercial porcelain insulators was monitored at an initial moisture level of 15–16%. A typical conventional process requires 40–50 h for drying a porcelain disk insulator of $\sim$300 mm diameter, while microwave drying in 6 kW furnace needs 12–15 h 0.4 kW Microwave power. The most important point to be noted here is that the drying rate was followed in a similar manner as observed for small samples. In each case, the surface temperature of the bodies was maintained at $\sim$58–65°C in order to avoid any catastrophic failure during final stage of drying. The slope of the drying curve for commercial insulators matches with that of small component drying. The results indicate that this can be commercially used to dry with out any crack in a reduced time compared with long conventional drying schedule.

Summary and Conclusions

The microwave drying of electroporcelain insulator components was carried out and the reliability of the process was compared with that of conventional drying process. It was observed that the microwave-dried samples displayed better bend strength and higher Weibull modulus values compared with their conventionally processed counterparts. Similar results were also observed in the Modulus measurement of these samples in green condition. These results and analysis indicate that fast microwave drying can be easily used for large scale drying of ceramic components.

Acknowledgments

The authors acknowledge the valuable suggestions by Shri G. Swaminathan, Head of Ceramic Technological Institute during the course of the investigation. The authors also acknowledge the support and permission of BHEL management to publish this work.

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