Use of Optical Fiber Sensor and Design of Experiments Statistical Methodology for Improving Dye Retention in a Quartz Ceramic Porous Material.

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Abstract

The fabrication process parameters of a quartz ceramic porous material are deliberately varied, and its dye retention after filtering an aqueous solution is evaluated with an optical fiber sensor. The parameters are analyzed in order of improving the retention, and determining the best fabrication conditions. According to the results, a reduction of 16.25% in relative dye concentration was obtained for the material sintered under 850°C for 120 minutes.

Key words:

Design of Experiments, Dye Retention, Quartz Ceramic Material.

Introduction

The factorial design of experiments is a statistical technique useful for the study of an optimal condition. It consists in a group of experiments organized in such a way that many process factors can be studied simultaneously, providing information about their interactions and synergetic effects. The process parameters are deliberately varied, and each factor is varied between two levels. A specific combination of factors is denominated a treatment, and the result that is measured is called response variable¹.

The factorial design was used for determining the best process parameters for the production of a ceramic porous material, made from α -quartz, designed for water treatment. The time of ultrasound treatment, and the time and temperature of the sintering process were varied, and a solution of water and food dye (Dr.Oetker® Strawberry – Color Index 16185) was passed through the material for evaluating its filtering capability.

Results and Discussion

Quartz powder obtained by milling of natural quartz was pressed under 30 MPa and sintered under $\sim 1000^\circ\text{C}$ for producing 4.35 mm thickness ceramic materials. The scanning electron microscopy analysis (SEM), shown in Figure 1, revealed a high porosity material and the X-Ray diffraction measurement indicated the α -quartz structure. Next, the material was tested on the retention of commercial food coloring.

Since the refractive index of solutions varies with the dye concentration, the samples could be analyzed by optical fiber sensor, in which the reflected light at the fiber-liquid interface is related to the sample refractive index, according to the Fresnel equation². The sensor was calibrated for the dye diluted solutions, and then tested for the filtered samples. The relative difference of concentration before and after the filtering was taken as the response variable. The results were analyzed with Minitab®17 Statistical Software.

A first full factorial experiment was carried out in order to evaluate if one of the three factors had no significant importance in the result. The result was that the ultrasound treatment had no significant effect, so it was not tested in the second factorial. The second factorial is shown in Chart 1. A reduction of 16.25% in relative dye concentration was obtained for the material sintered under 850°C for 120 minutes.

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Image 1. Material pressed and sintered and its SEM image, showing pores with diameters varying from 960 nm to 2.8 μm .

Chart 1. Second factorial experiment.

Experiment	Sintering temperature ($^\circ\text{C}$)	Sintering time (min)	Relative concentration reduction (%)
B21	850	60	15,76
B22	850	120	16,25
B23	950	60	12,43
B24	950	120	14,03

Conclusions

The methodology of the full factorial experiment was effective for finding a better operating condition for the manufacturing of the ceramic material. The reduction in dye concentration is significant, since it is commonly necessary to use high-tech processes in order to produce materials capable of retaining such small structures³.

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