# Optimization of Catalyst Preparation using High-Throughput Techniques

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## Introduction

Zeolites are in common use as solid acid catalysts in many commercial processes. The strength and distribution of acid sites is often a key determinant of the activity and selectivity of the catalyst. This can be controlled either by direct synthesis or by post-synthesis treatments, of which steaming is among the most frequently used. Optimization of the steaming conditions is of critical importance to attain acid site balance for the best possible catalyst performance, and a detailed understanding of the relationship between treatment conditions, acid site distribution and catalyst performance is essential for good control of this step of the manufacturing process. The use of high-throughput techniques enables preparation, testing and characterization of large numbers of samples, providing a detailed mapping of catalyst response to these variables and a more complete picture of the changes in catalyst properties.

## Materials and Methods

A number of different bound zeolite samples were studied, including MFI, FAU, and MOR. Catalysts were steamed in a Heat Treatment Unit (HTU)[1]. This unit allows treatment of 48 samples at a time at various combinations of time, temperature and steam level. Testing was carried out using a Reactor Assay Module[2], which allows testing of samples 48 at a time. Catalysts were tested by cracking of n-heptane at various conditions.

## Results and Discussion

Contour maps showing the response of n-heptane conversion over MFI catalysts to variations in steaming conditions are shown in Figure 1. These maps make it easy to see the response of catalyst activity to steaming conditions and to identify the correct range of conditions necessary to obtain the desired catalyst activity. It can be seen that the gradients become much narrower as steam level is increased, showing that tighter control of steaming conditions is necessary at higher steam levels. Similar maps were obtained for other zeolites studied.

Acid site distribution for selected samples was studied using $NH_3$-TPD. These data show large differences between fresh and steamed samples, especially in the number of strong acid sites (those desorbing $NH_3$ above 350°C). It is the change in the number of these strong acid sites which most strongly correlates with changes in catalyst activity.

| | moles $NH_3$/g | | | n-heptane |
|---|---|---|---|---|
| | 250-350°C | 350-500°C | total | Conversion |
| Fresh | 0.010 | 0.199 | 0.209 | |
| 650°C, 4 hr, 27% stm. | 0.006 | 0.063 | 0.069 | 70% |
| 650°C, 8 hr, 60% stm. | 0.007 | 0.056 | 0.063 | 39% |

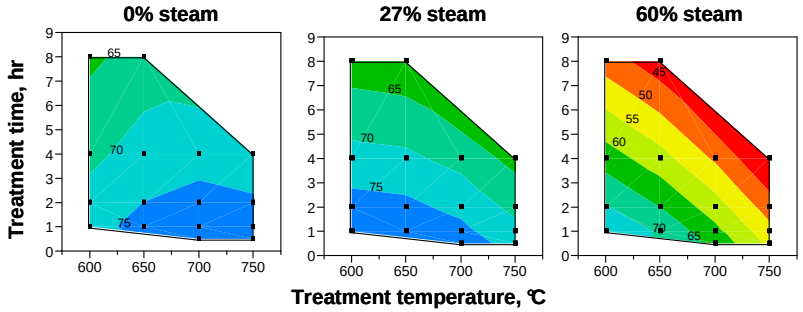


**Figure 1. Response surface maps of n-heptane conversion over MFI (283°C, 3.9 WHSV).**

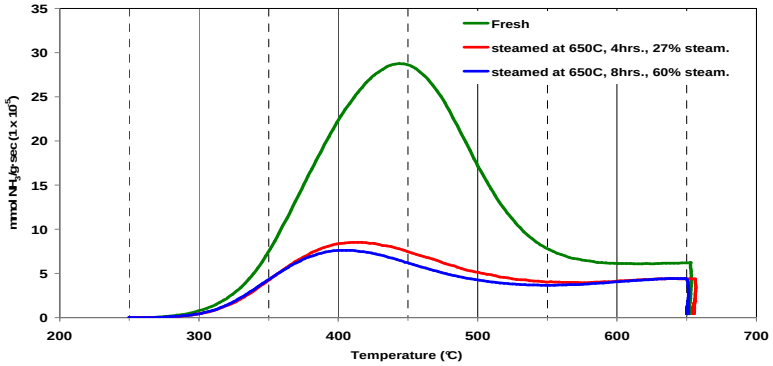


**Figure 2. $NH_3$-TPD of selected MFI samples**

## Significance

This work can not only lead to improved control of manufacturing, but also to a better understanding of the role of acid site distribution.

## References

1. ML Bricker, L Bauer, MG Gatter, J Abrahamian, RD Gillespie in "Combi and HT Discovery and Optimization of Catalysts and Materials" (Potyrailo & Maier, eds) Chapter 6: "Use of Combinatorial Heat Treatments to accelerate the commercialization of materials for use in catalysis." CRC press June, 2006.
2. ML Bricker, RD Gillespie, JS Holmgren, JWA Sachtler, RR Willis in "High Throughput Analysis: A tool for Combinatorial Material Science". Chaper 26: "Scaling-up of Catalysts Discovered from Small scale experiments". . Kluwer Academic/Plenum Publishers November, 2003 ISBN0-306-47758-0.