

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In <i>Inter Partes</i> Reexamination of:	)	
	:	Examiner: DIAMOND, ALAN D
BULL ET AL.	)	
	:	Group Art Unit: 3991
Reexamination Control No. 95/001,453	)	
	:	Confirmation No: 2755
Patent No. 7,601,662	)	
	:	
Issued: October 13, 2009	)	
	:	
For: COPPER CHA	)	
ZEOLITE CATALYSTS	)	
	:	

**Mail Stop Inter Partes Reexam**  
Central Reexamination Unit  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**SECOND DECLARATION OF AHMAD MOINI, PH.D., UNDER 37 C.F.R. § 1.132**

I, Ahmad Moini, do declare and say as follows:

1. I am a Senior Research Associate for BASF Corporation, the successor in interest to Engelhard Corporation and BASF Catalysts LLC (BASF), the owner of United States patent number 7,601,662 ("the '662 patent"). I received a B.S. in Chemistry from Eastern Washington University in 1982 and a Ph.D. in Chemistry from Texas A&M University in 1986. I have been a scientist at BASF Corporation. I am a co-inventor of the subject matter described and claimed in the '662 patent.

2. I have been involved in heterogeneous catalyst research for more than 20 years, first as a researcher at Mobil Research and Development Corporation, and later at BASF Corporation. I am a named inventor on 33 granted United States patents, some of which pertain to zeolites for use as automotive catalysts.

3. I submitted a first declaration in this matter on February 9, 2011. I have been asked to conduct several experiments to compare the catalytic performance of the catalysts claimed in the '662 to the performance of catalysts that have been considered the closest prior art in this reexamination. I have reviewed the '662 patent, the Office Action dated November 16, 2010, the Action Closing Prosecution dated November 18, 2011, including the documents cited in the actions,

as well as additional documents cited in this second declaration. I have read Dr. Lercher's declaration and Exhibit C-4.

#### **Dedecek Samples**

4. It is my understanding that the Examiner finds that with respect to the rejections based on Dedecek in view of Chung, the closest prior art is two examples from Dedecek, one being the natural CuCHA zeolite having a Cu/Al atomic ratio of 0.38 (the sixth entry in Table 2 on page 66) and other being the synthetic CuCHA zeolite having a Cu/Al atomic ratio of 0.32 (the eleventh entry in Table 2 on page 66). I have been asked to recreate these examples from the Dedecek reference.

#### **Natural CHA Sample:**

5. For the natural CuNatCHA example, the Dedecek reference started with natural chabazite from North Korea having the chemical composition 63.89% SiO<sub>2</sub>, 17.48% Al<sub>2</sub>O<sub>3</sub>, 8.37% Fe<sub>2</sub>O<sub>3</sub>, 5.15% K<sub>2</sub>O, 3.10% CaO, 1.21% MgO, 0.40% TiO<sub>2</sub> and 0.39% Na<sub>2</sub>O. However, due to import restriction under the United States Department of Treasury Office of Foreign Assets Control, we are not able to obtain natural chabazite samples from North Korea. Accordingly, I obtained a sample of natural chabazite zeolite sample from a mine in Bowie, Arizona as an indirect comparison to the CuNatCHA sample in Dedecek. The Bowie sample is comparable to the one from North Korea in terms of the metal oxides content other than silica or alumina. The chemical composition of the Bowie sample, determined by X-ray Fluorescence, was 64.74% SiO<sub>2</sub>, 21.54% Al<sub>2</sub>O<sub>3</sub>, 9.84% Na<sub>2</sub>O, 1.37% Fe<sub>2</sub>O<sub>3</sub>, 0.93% K<sub>2</sub>O, 0.59% MgO, and other residual components. The primary phase of this material was determined to be Chabazite, with secondary mineral phases present, as would be expected by a natural material.

6. Per the example preparation in Dedecek, the CuNatCHA sample was equilibrated three times with 0.5M NaCl (20 ml of solution per 1 g zeolite) for 2 hours, and a fourth was carried out overnight. These equilibration times varied from Dedecek's procedure, but it is understood that, with this type of alkali equilibration, the key parameter is the number of exchanges rather than the total exchange time. After the ion exchange, the Na-chabazite was washed with distilled water and dried at room temperature. The powder from the NaCl treatment was subjected to two Cu acetate treatments, under the same conditions described in Dedecek et al., Table 2, entry on row 6. The final product was washed thoroughly with distilled deionized water. Elemental analysis revealed 65.13%

SiO<sub>2</sub>, 17.45% Al<sub>2</sub>O<sub>3</sub>, 8.91% CuO, 4.52% Na<sub>2</sub>O, 3.31% Fe<sub>2</sub>O<sub>3</sub>, 0.66% K<sub>2</sub>O, 0.44% MgO. Based on these data, the Cu/Al ratio was 0.32.

Synthetic sample:

7. A sample of synthetic Chabazite was synthesized according to the procedure referenced in Dědeček et al. Zeolite Y (CBV-600) was mixed with KOH solution and reacted at 95°C for 92 hours. The resulting product was washed thoroughly with distilled deionized water. The formation of Chabazite product was confirmed by XRD.

8. The chabazite powder was subjected to four NaCl equilibrations. The first three treatments were for two hours, while the fourth was carried out overnight. These equilibration times varied from Dedecek's procedure, but it is understood that, with this type of alkali equilibration, the key parameter is the number of exchanges rather than the total exchange time. The resulting powder was washed thoroughly with distilled deionized water.

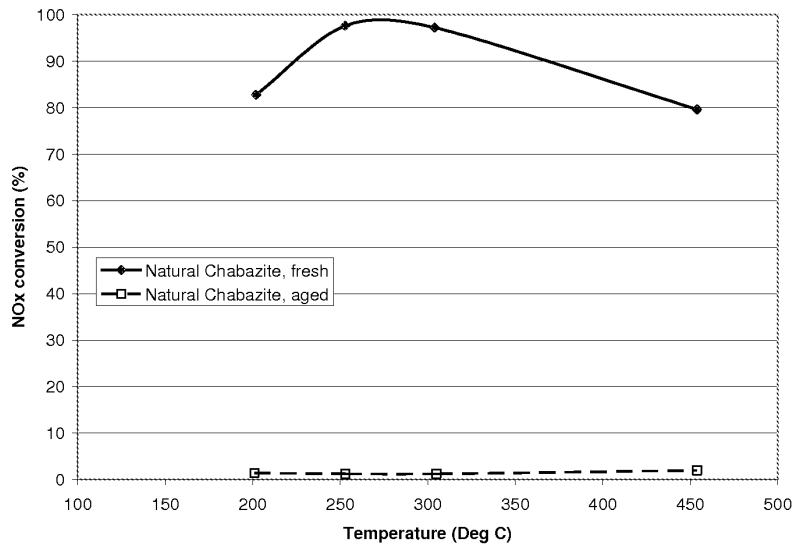
9. The powder from the NaCl treatment was subjected to two Cu acetate treatments, under the same conditions described in Dědeček et al., Table 2, entry on row 11. The final product was washed thoroughly with distilled deionized water. Elemental analysis revealed 58.47% SiO<sub>2</sub>, 22.16% Al<sub>2</sub>O<sub>3</sub>, 11.4% CuO, 5.17% K<sub>2</sub>O, 2.57% Na<sub>2</sub>O. Based on these data, the Cu/Al ratio was 0.33.

NOx Conversion Testing:

10. The natural and synthetic samples made in accordance with paragraph 5-9 above were tested for fresh and aged NOx conversion. Each catalyst sample was disposed on a 1 inch diameter X 3 inch long cellular ceramic core having a cell density of 400 cells per square inch and a wall thickness of 6 mil at a catalyst loading that was in the range of 2.28 and 2.52 g/in<sup>3</sup>. They were then tested for nitrogen oxides selective catalytic reduction efficiency and selectivity by adding a feed gas mixture of 500 ppm of NO, 500 ppm of NH<sub>3</sub>, 10% O<sub>2</sub>, 5% H<sub>2</sub>O, balanced with N<sub>2</sub> to a steady state reactor containing the catalyst core at a space velocity of 80,000 hr<sup>-1</sup> across a 150 °C to 460 °C temperature range.

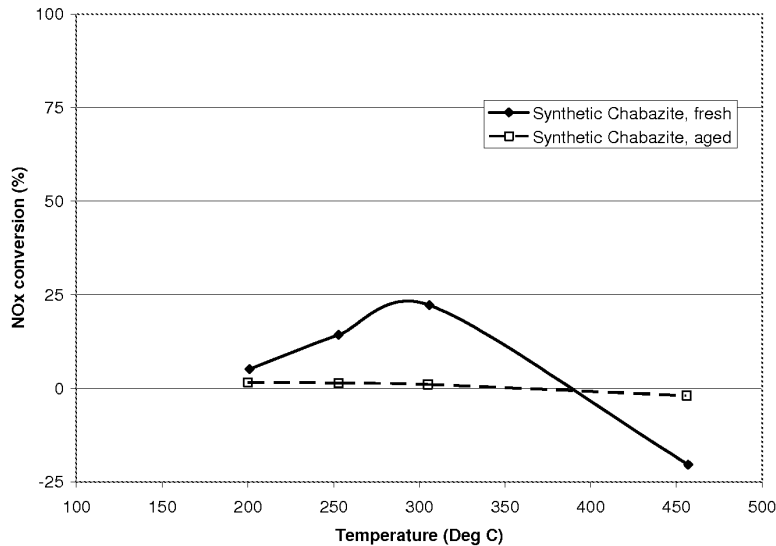
11. The samples were then hydrothermally aged at 850 °C for six hours. The samples were then each tested again for NOx conversion using the same parameters as for fresh. A plot of the fresh and

aged conversion is shown below for the natural chabazite:



12. As shown in the plot, after aging, the NOx conversion of the natural chabazite was destroyed across the entire temperature range. This material would not be a material of interest as an ammonia SCR catalyst, as the fresh conversion is useless if the catalyst can not survive hydrothermal aging and maintain high conversion over a temperature range of 200 to 450 °C. The excellent fresh and aged performance of the catalyst claimed in the '662 patent is quite unexpected in comparison to these results.

13. A plot of the fresh and aged conversion of the synthetic sample prepared according to paragraphs 7-9 is shown below:



14. This material showed extremely poor fresh and aged conversion. Aging destroyed the NOx conversion of the material. It should be noted that both the fresh and aged samples show negative NOx conversion at approximately 450°C. Instead of converting NOx to nitrogen, these samples produce NOx at these temperatures. This would not be a material of interest as an ammonia SCR catalyst. The excellent fresh and aged performance of the catalyst claimed in the '662 patent is quite unexpected in comparison to these results.

#### Zones Example 1

15. I have reviewed the Zones reference, which discloses only one working example. Examples 1-4 show synthesis of a SSZ-62 sample which was then calcined and underwent NH<sub>4</sub> exchange, and ultimately to the proton form, H<sup>+</sup>-CHA with a silica to alumina ratio of 22.

16. I located data for an H<sup>+</sup>-CHA sample that was tested for NOx conversion using ammonia SCR. The testing was performed by disposing the catalyst on a 1 inch diameter X 3 inch long cellular ceramic core having a cell density of 400 cells per square inch and a wall thickness of 6 mil at a catalyst loading of 2.13 g/in<sup>3</sup>.

17. The sample was tested for nitrogen oxides selective catalytic reduction efficiency and selectivity by adding a feed gas mixture of 500 ppm of NO, 500 ppm of NH<sub>3</sub>, 10% O<sub>2</sub>, 5% H<sub>2</sub>O, balanced with N<sub>2</sub> to a steady state reactor containing the catalyst core at a space velocity of 80,000

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