



## UV-visible characterization of gold exchanged in beta zeolite

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The gold/zeolite system was prepared by ion exchange method. The gold introduced into Beta zeolite and reduced at various temperatures by hydrogen was studied by means of UV-Visible diffuse reflectance spectroscopy. Three types of gold species were observed in the samples: cations, clusters and nanoparticles.

### 1 Introduction

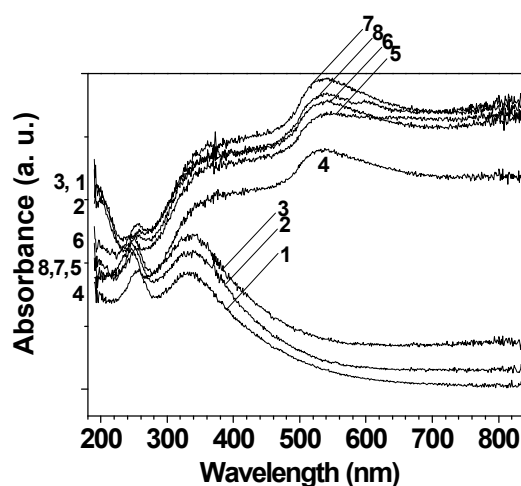
Gold deposited on metal oxides has been reported as active catalyst for many reactions [1]. Usually, gold exhibits activity when the size of its nanoparticles is less than 5 nm [2]. The stabilization of small metal particles and their activity strongly depend on preparation method and on support used [1]. Zeolite material possesses adjustable acidic properties and regular molecular-size channels in the crystalline lattice. These features are to be able to provide inclusion of metal ions into zeolite matrix with subsequent transformation to ultrafine metal particles and clusters [3, 4]. In the present paper, the effect of changing of reduction temperature and concentration on the state of gold in Beta zeolite were studied.

### 2 Experimental

A protonated form of Beta-zeolite with SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio equal to 20 was used for sample preparation. The gold concentration in prepared samples was analyzed by energy dispersive spectroscopy and state of gold was studied by UV-visible diffuse reflectance spectroscopy. The spectra

presented in Figs.1 and 2 were obtained by subtraction of the support spectrum from the spectra of Au supported samples.

The solution of [Au(NH<sub>3</sub>)<sub>4</sub>](NO<sub>3</sub>)<sub>3</sub> complex of two different concentrations for ion exchange was prepared by reac-

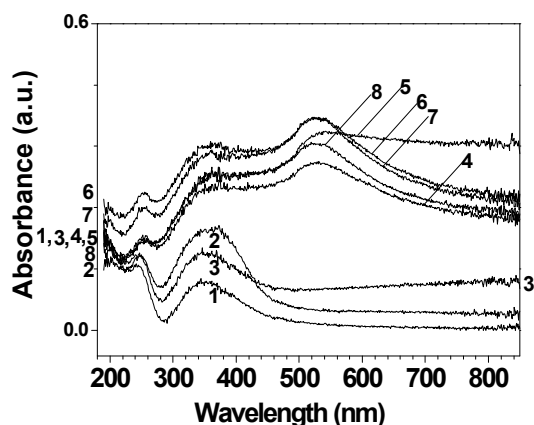


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**Figure 1.** UV-Visible spectra of 1 wt.% Au/Beta before reduction (1) and reduced at: 25°C (2), 50°C (3), 100 °C (4), 200 °C (5), 300 °C (6), 400 °C (7), 500 °C (8).

tion of  $\text{HAuCl}_4$  with  $\text{NH}_4\text{OH}$  [5]. After ion exchange the samples were reduced by  $\text{H}_2$  at different temperatures. The final concentrations of gold in the two samples measured by



**Figure 2.** UV-Visible spectra of 0.5 wt.% Au/Beta before reduction (1) and reduced at: 25°C (2), 50°C (3), 100 °C (4), 200 °C (5), 300 °C (6), 400 °C (7), 500 °C (8).

energy dispersive spectroscopy were approximately 0.5 and 1.5 wt. %.

### 3 Results and discussion

Several absorption bands of the ion exchanged form and reduced gold samples were detected with UV-Visible spectroscopy (Figs. 1 and 2). The band at  $\lambda=190$  nm is attributed to the  $\text{Au}^{1+}$  ion according to reference data [6, 7]; the features at  $\lambda=250$  and at 340 nm could be assigned to  $\text{Au}_n^{\text{m}+}$  clusters [8]; the peak with maximum at  $\lambda=550$  nm is assigned to the surface plasmon resonance of Au nanoparticles

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[6, 7] located on external surface of zeolite microcrystals. Increase of reduction temperature up to the 100°C (Figs. 1 and 2) leads to decrease of relative intensity of the peak associated with  $\text{Au}^{1+}$  and to rise of relative intensity of the plasmon resonance due to the nanoparticles. Further temperature increase does not reveal significant changes in the spectra.

Spectra for samples with different gold concentrations are similar except relative intensities, which rise with gold concentration. The sample with the lower concentration of gold shows higher contribution of gold species at 350 nm (which could be interpreted like clusters) than the samples with higher concentration.

### 4 Conclusions

Gold clusters and nanoparticles were produced by means of the ion-exchange from aqueous solution followed by hydrogen reduction in Beta-zeolite matrix. The method is similar to that for Ag; however, the complexation of Au precursor provides appearance the cluster-like species at the ion exchange step. Au clusters and nanoparticles were detected by the optical features: the band of cations with the maximum in the range 190 nm, clusters in the region 280 and 340 nm and the plasmon resonance band in the region about 550 nm. The contribution of various Au species in the reduced samples can be regulated by concentration of gold in beta zeolite and reduction temperature. The Beta zeolite with the reduced gold species is of interest for catalysis application.

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