

Investigation of the surface interactions of indole with mesoporous silica using FTIR spectroscopy and hyperspectral imaging

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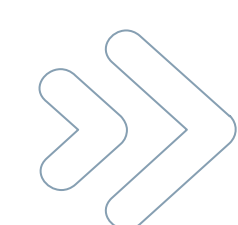
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INTRODUCTION

The adsorption of indole from carbon tetrachloride solution on mesoporous silica (MS) material was investigated by kinetic FTIR spectroscopic measurements. This result is correlated to the capability of indole N-H to participate in hydrogen bonding with the MS surface. Results of FTIR spectroscopy evidence the presence of indole adsorbed on the surface of MS while the analysis of hyperspectral images reveals the possibility of constructing effective qualitative hyperspectral sensors for indole based on MS as a sensing substrate.



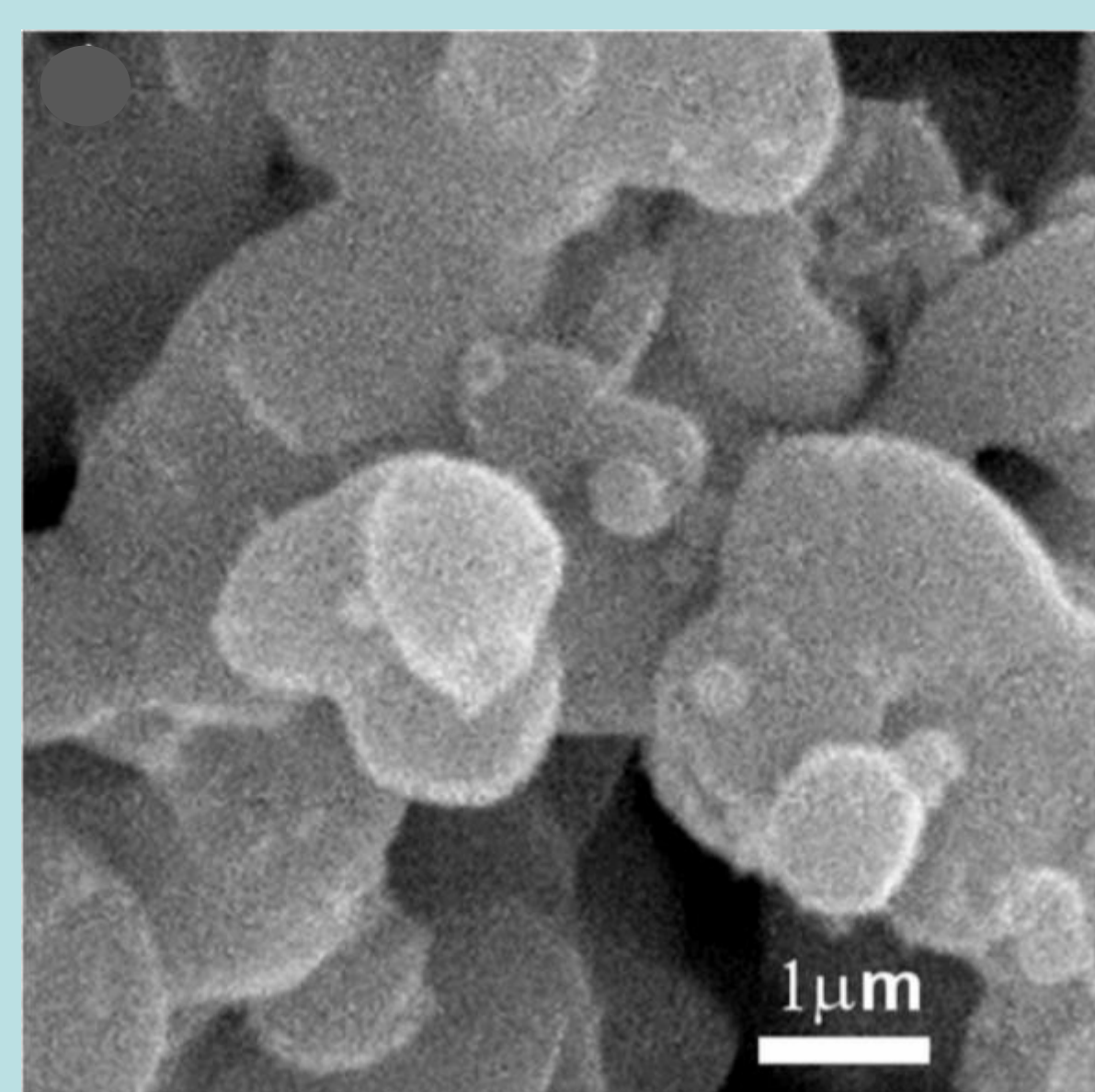
EXPERIMENTS

The adsorption kinetics of the amides from CCl₄ solution were recorded directly, namely the cuvette with the amide solution was mounted on a magnetic stirrer and FTIR spectra were continuously recorded while stirring.

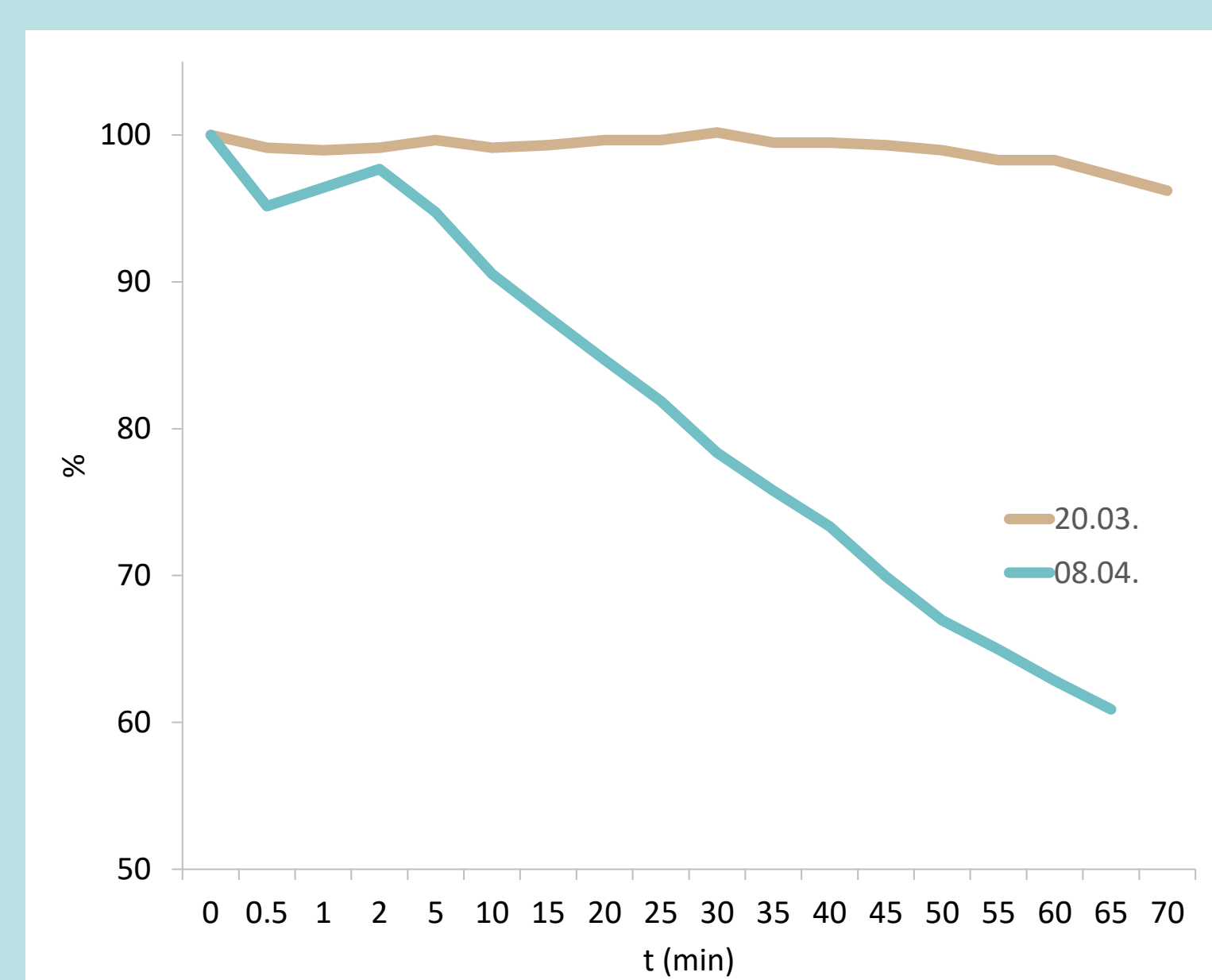
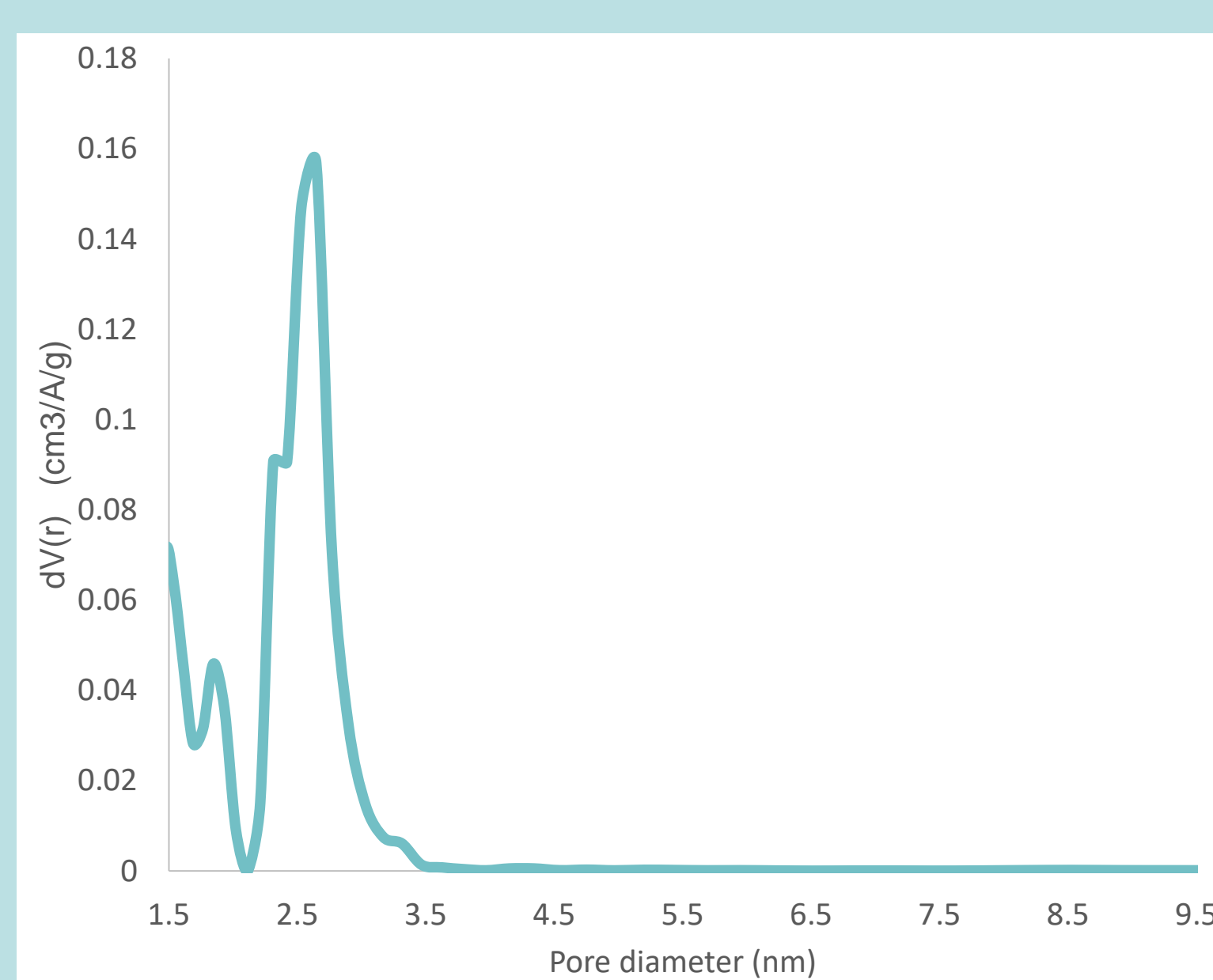
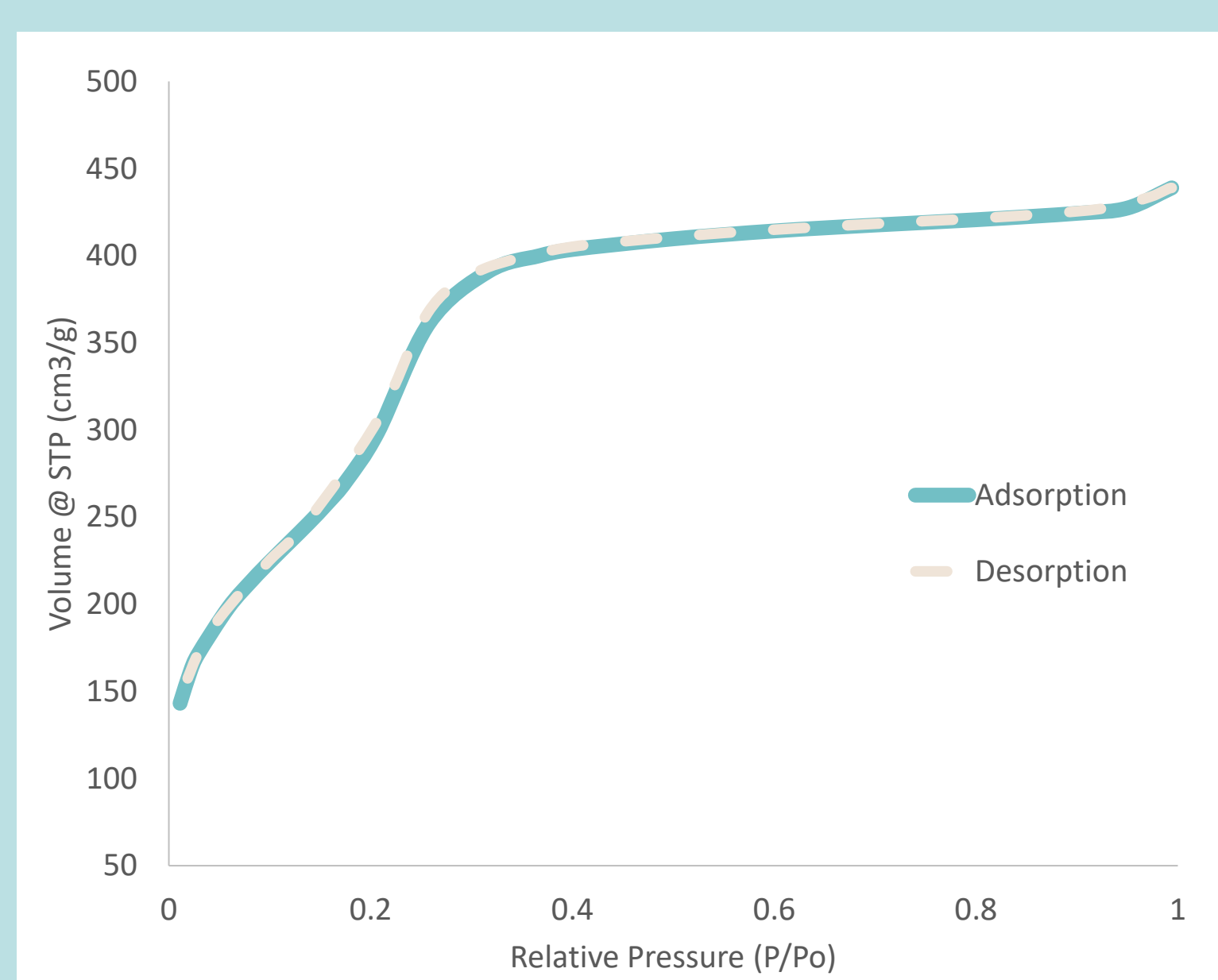
For characterization of indole on the surface of the MS, 300 mg of MS was added to the amide solutions at a concentration of 0.02 M, the solutions were stirred on a magnetic stirrer at room temperature for 1 h. After stirring, the solvent was evaporated, and the obtained MS materials were further characterized by FTIR and hyperspectral measurements.



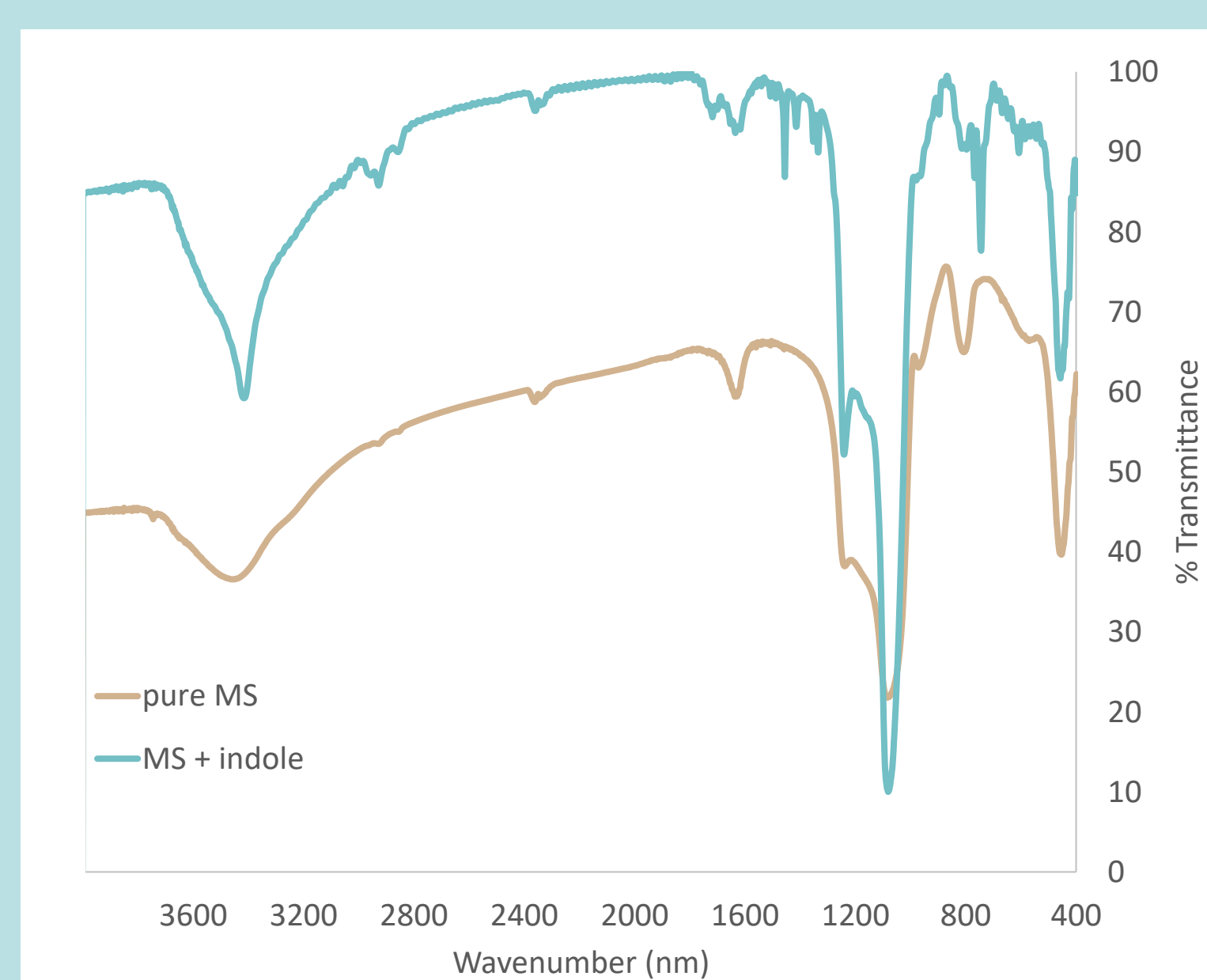
RESULTS AND DISCUSION



Mesoporous silica material was synthesized by condensation of silica precursor (tetraethoxysilane) in basic aqueous environment, in the presence of cetyltrimethylammonium bromide (CTAB), as a mesopore template. The Brunauer, Emmett and Teller (BET) specific surface area of the material after removal of the CTAB template by calcination was 1053 m²/g with predominant pore size of 2.6 nm, calculated by the Barrett, Joyner and Halenda (BJH) method. The obtained BET isotherm for MS is type IV, typical for mesoporous silica materials, without hysteresis. Zeta potential value of the MS material is -35 mV.

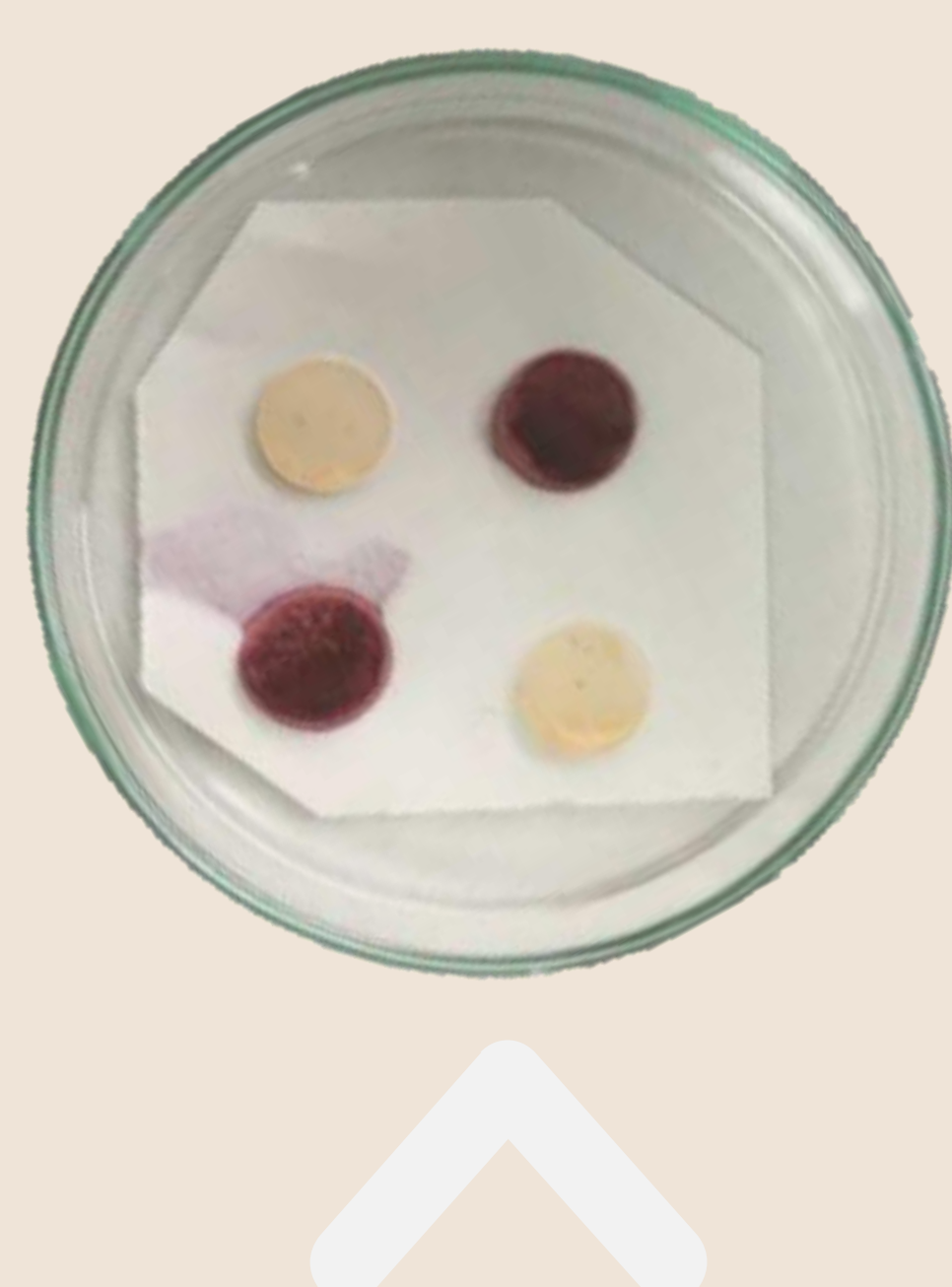


Adsorption at different time intervals, with repeated experiments after 3 weeks significant differences in indole adsorption of both higher and lower concentrations are observed. It can be assumed that the reason for the different adsorption capacities of indole is the highly altered surface of mesoporous silicate. Namely, mesoporous silicate adsorbs moisture from the air and can therefore significantly reduce the capacity towards some specific adsorbates.

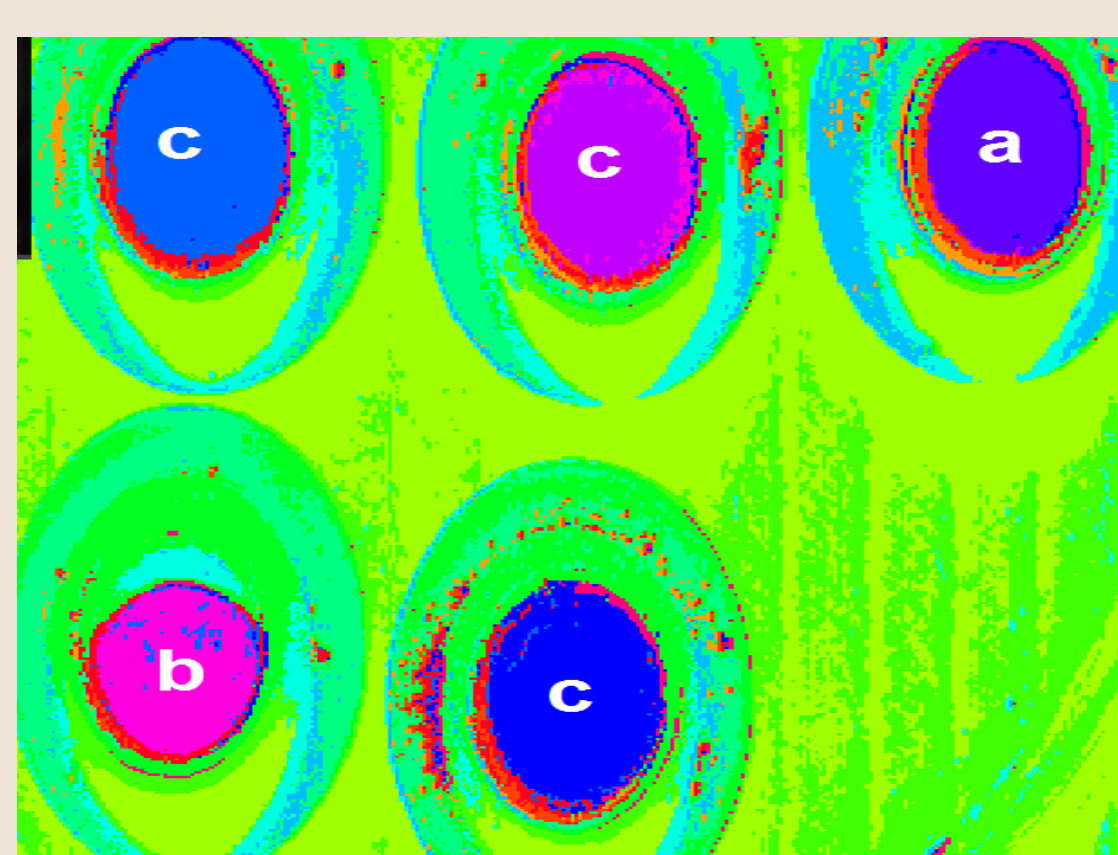


By analyzing the FTIR spectrum of pure MS, five characteristic bands for silicate materials can be observed at wavenumbers 3440, 1630, 1080, 960 and 802 cm⁻¹. In case of the FTIR spectra of MS with the adsorbed indole, additional bands can be observed. The characteristic vibrational and deformational N-H indole bands can be observed at 3415, 1465, 1414 and 1241 cm⁻¹. The characteristic aromatic ring stretching, and deformation bands can be seen at 768 and 745 cm⁻¹.

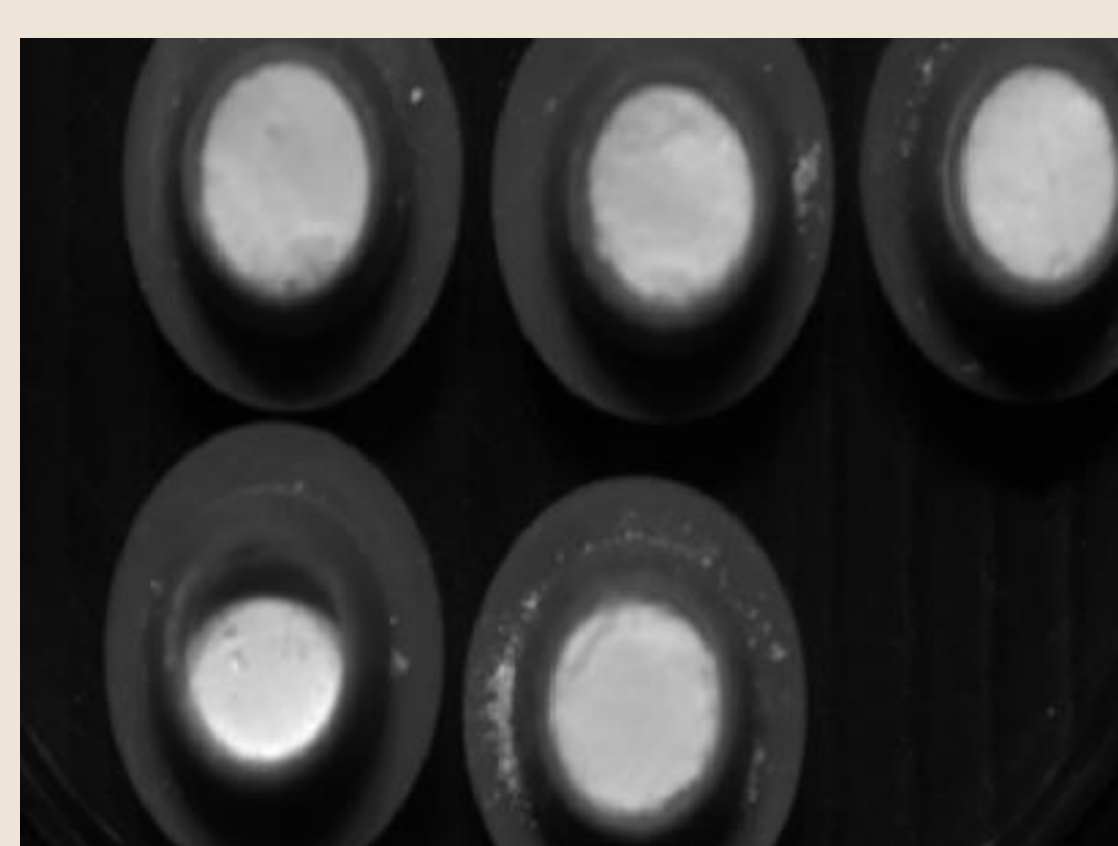
- a) Pure MSN
- b) Indole adsorbed on MSN
- c) Amides adsorbed on MSN



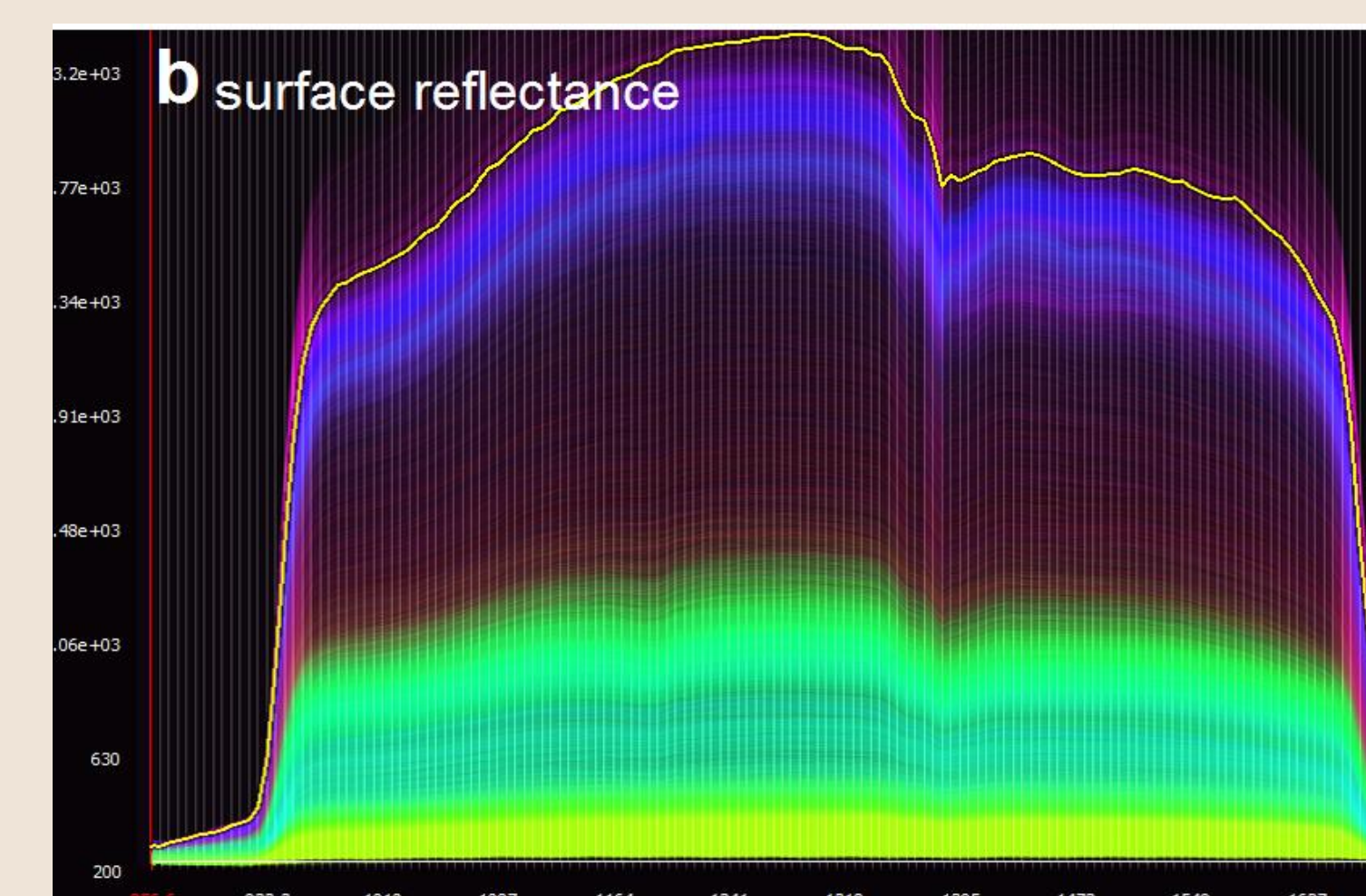
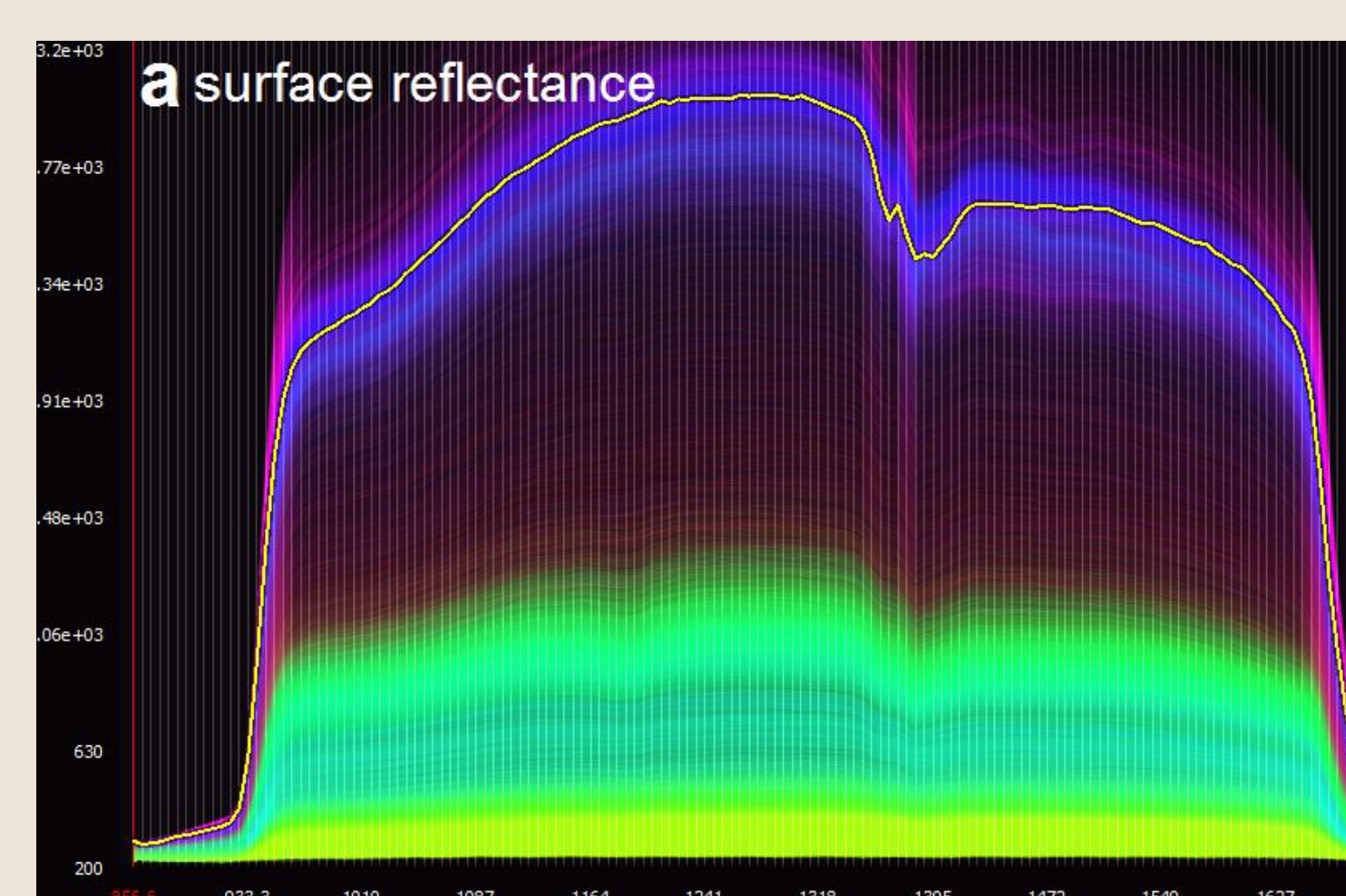
Proof by Ehrlich's reaction



Hyperspectral Image after pixel clustering



RGB Image



Similar to the analysis in the MIR region by FTIR spectroscopy, also in the near NIR region, significant changes in the reflectance in the range of N-H overtone vibrations (1310-1400nm) can be observed in hyperspectral photographs.

Based on the difference in the values of selected PCA components, indole and different small biomolecules (amides) on the surface of mesoporous silicate materials can be clearly distinguished by utilizing the employed clustering method. The changes observed in the hyperspectral responses and their derivatives can be of significant practical use for the development of new qualitative sensors for small bioactive molecules as indole.

B. Jović, M. Panić, N. Radnović, K. Zivojević, M. Mladenović, V. Crnojević, N. Knežević.
Investigation of the surface interactions of selected amides with mesoporous silica using FTIR spectroscopy and hyperspectral imaging, Journal of Molecular Structure 1219 (2020) 128562

