

PREPARATION AND STUDY OF NiO-TiO₂ MIXED CATALYSTS

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INTRODUCTION

Catalysis is an integral part of chemistry, chemical technology and industry. Many important chemical compounds and industrial products are produced via catalytic reactions.

Our work was part of the research on catalytic systems composed of different metal oxides, pursued at the department of nuclear chemistry, CTU FNSPE. We participated in preparation and study of catalytic properties of mixed oxide catalysts NiO-TiO₂ with varying ratio of both components. These components had been chosen because of their properties. NiO is important catalyst of many chemical reactions and TiO₂ is widely used photocatalyst. Mixing them could yield a two-component catalyst featuring combined interesting properties of both components.

2. Preparation of catalysts

Precursors: tetra-n-butyl-orthotitanat and Ni(NO₃)₂

Chemical preparation:

- chemical conversion of tetra-n-butyl-orthotitanatu into Ti(NO₃)₄
- mixing of Ti(NO₃)₄ and Ni(NO₃)₂
- > of 15 samples with amount of both components varying in the range 0-100%
- dissolving of samples in nitrate acid
- > formation of mixture of Ti⁴⁺ + Ni²⁺ + NO₃⁻ + H₂O in acid solution
- > precipitation of mixture with potassium hydroxide
- filtration and drying - removing abundance of aqueous phase from the precipitate
- calcination of remaining sediment at temperature 450°C
- > formation of chemical mixture of crystals NiO-TiO₂
- homogenization of samples in spherical mill



Fig. 1: RTG diffraction device

3. Study of properties

Non-stoichiometric oxygen: iodometric titration

Size of specific surface area: selective adsorption of nitrogen from its mixture with hydrogen at low temperatures on the surface of measured sample

Phase composition and structure of lattice: RTG powder diffraction

Catalytic activity: Measurement of kinetics of catalytic decomposition of hydrogen peroxide at constant temperature and pressure

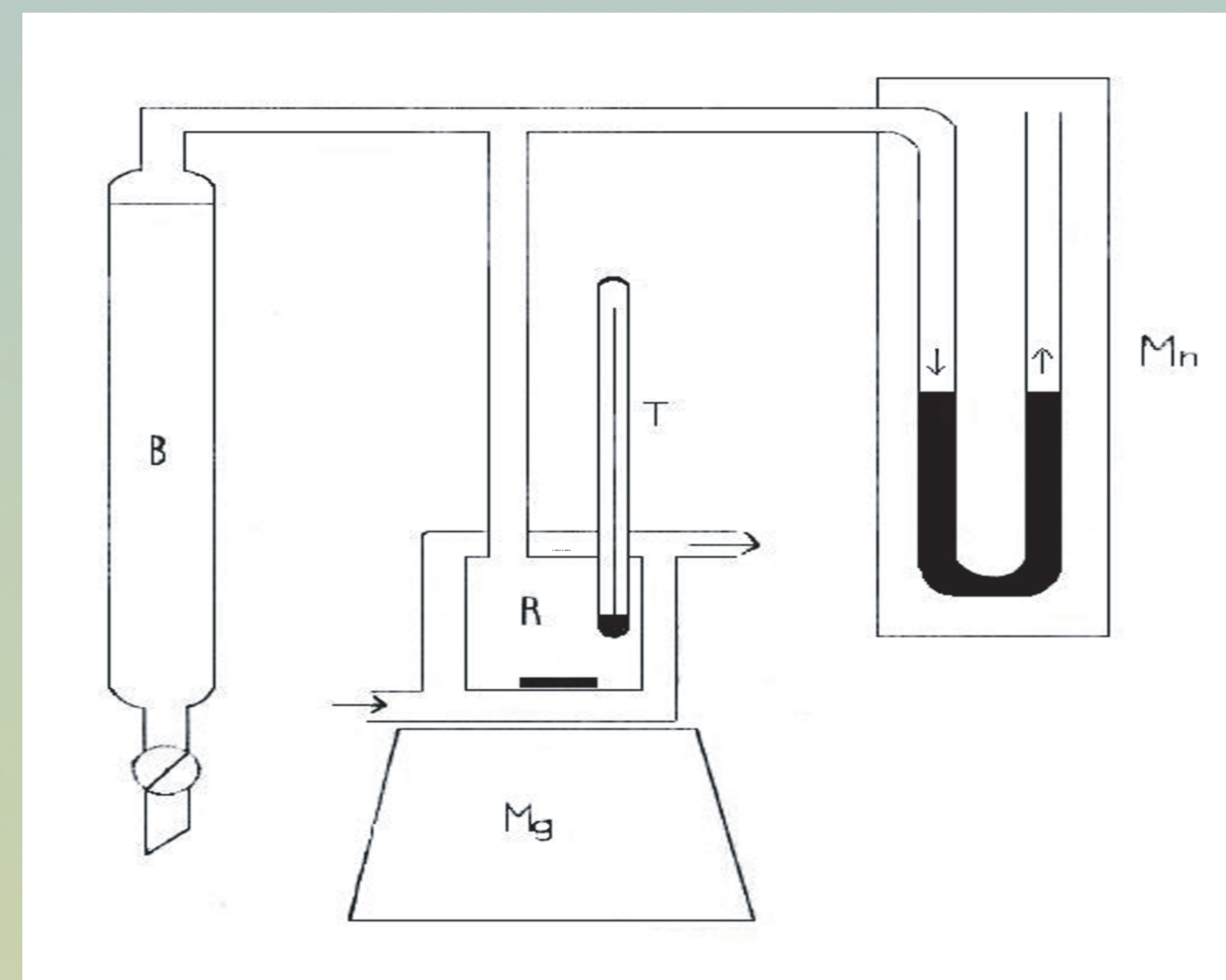


Fig. 2: Scheme of apparatus for catalytic activity measurement, where R is thermostatted reactor, T is thermometer, Mg is magnetic stirrer, B is buret and Mn is manometer

4. RESULTS

Non-stoichiometric oxygen was measured only in samples with significant abundance of NiO (>80%). Obtained results indicate that prepared TiO₂ is stoichiometric.

Dependency of specific surface area on composition is shown on figure 3. The curve is non-monotonous with maximums at 70 and 100% of TiO₂. Absolute size of surface is rather small, which may negatively impact the catalytic properties.

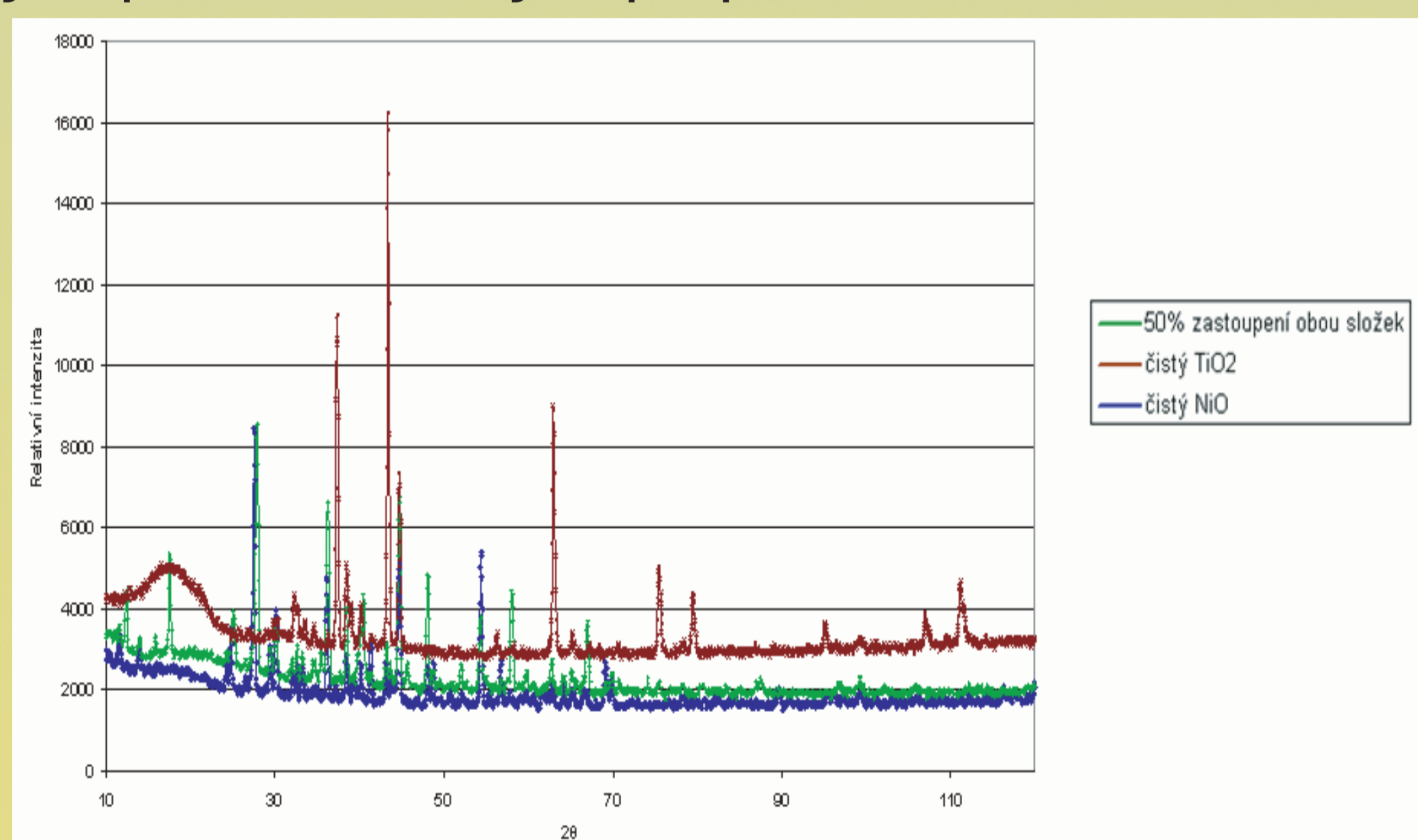


Fig. 4: Example of diffractogram

Measurement of kinetics of catalytic decomposition of hydrogen peroxide indicates, that catalytic activity of the system decreases with increasing amount of TiO₂ in the samples; values of virtual activation energy E_a were determined to be in the range of tens to hundreds kJ/mol. Figure 5 shows basic kinetic curve of catalytic reaction.

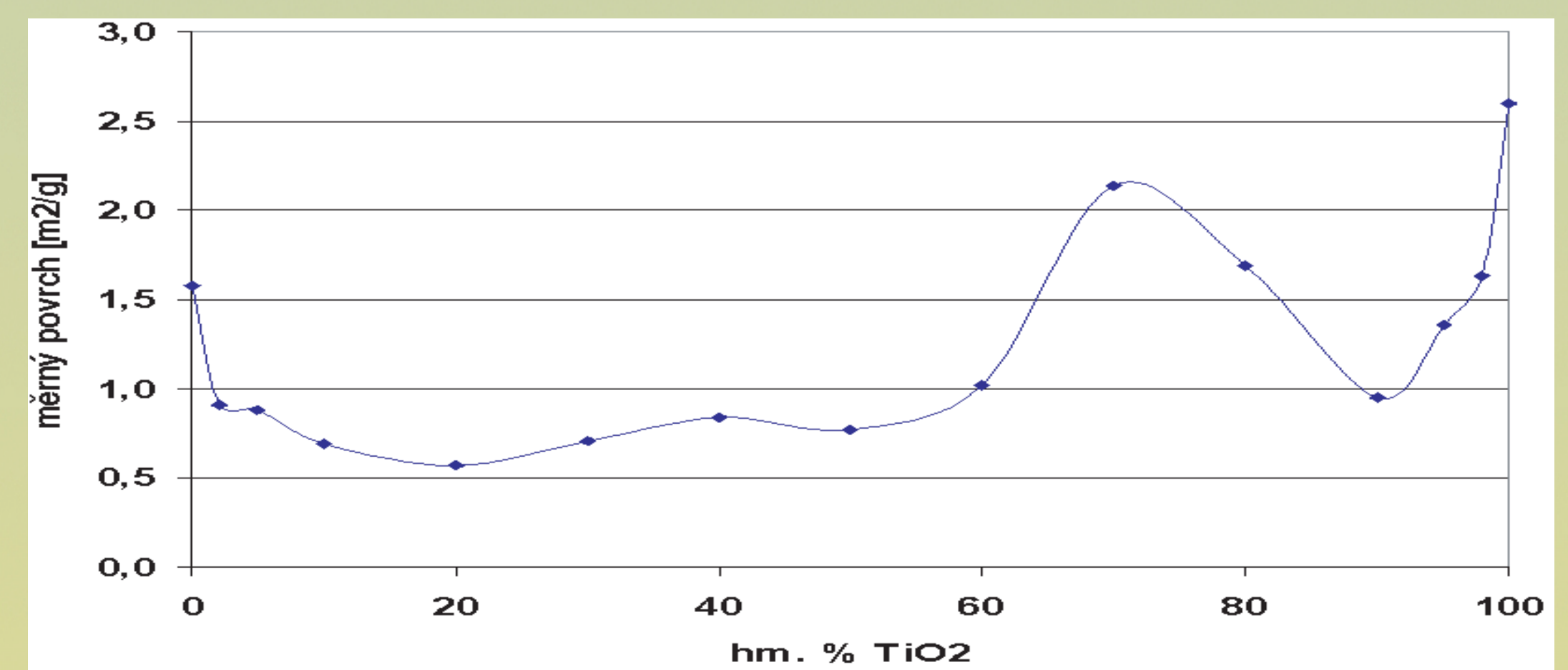


Fig. 3: Dependency of specific surface area on composition of samples

Based on obtained diffractograms, we can deduce that both compounds in all samples have crystalline character. Only diffraction lines of NiO and TiO₂ structures are present in the samples. Analysis of FWHM of selected peaks indicates that no formation of solid solution occurred in the system. Linear size of crystallites is in the range of tenths of micrometers. Example of diffractogram is shown in figure 4.

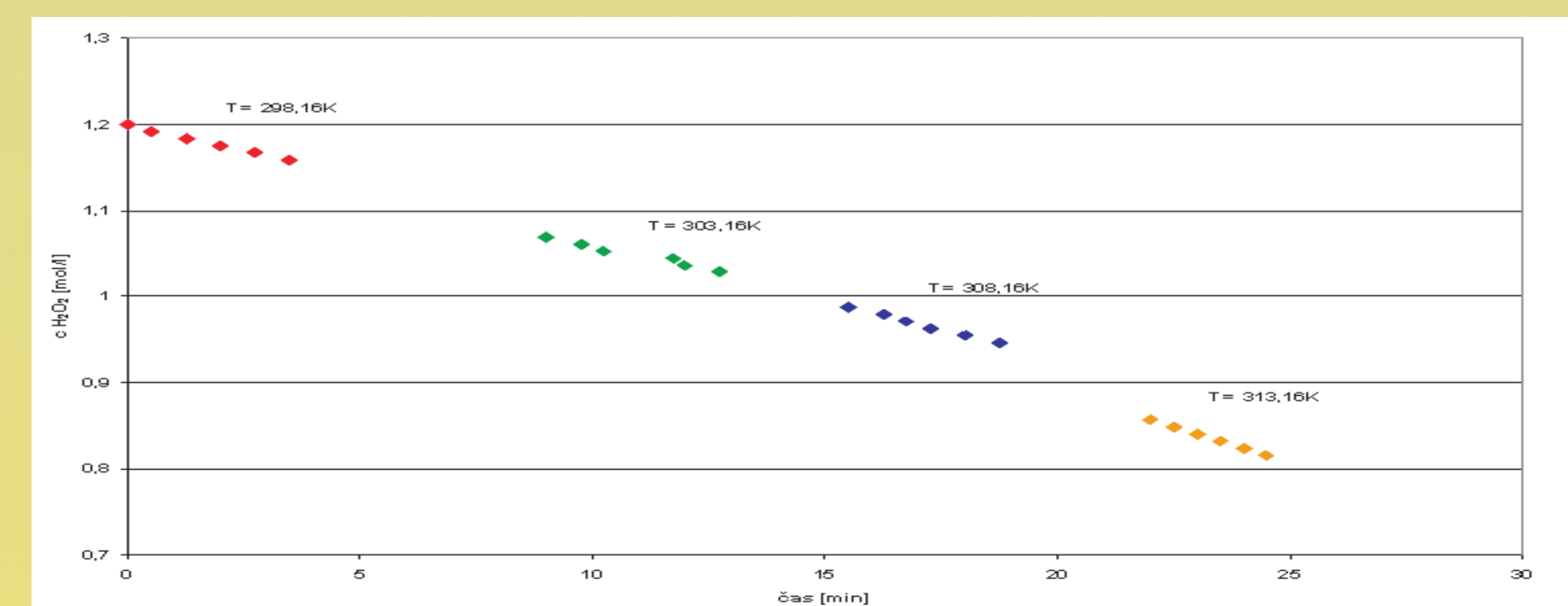


Fig. 5: Kinetics of decomposition of hydrogen peroxide at different temperatures

CONCLUSIONS

We prepared series of 15 samples of mixed catalysts with varying ratio of both compounds and determined some of their physico-chemical properties, including measurement of catalytic activity of basic unprocessed series.

Properties of prepared catalysts will be further studied after various processing, e.g. after implantation of accelerated foreign ions (N⁺, N²⁺) into the crystalline lattice of catalyst, irradiation of catalysts with high doses of gamma rays and accelerated electrons, chemical impregnation of surface, thermal processing, and after reduction of catalysts with hydrogen at high temperatures.