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OPTIMIZACIJA SONOKEMIJSKE SINTEZE NANOČESTICA ZrO_2

OPTIMIZATION OF SONOCHEMICAL SYNTHESIS OF ZrO_2 NANOPARTICLES

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INTRODUCTION

The dimensions and properties of nanoparticles enable their wide application. Today, various syntheses are being developed to obtain nanoparticles. Sonochemical synthesis resulting from ultrasonic cavitation [1, 2] is one of the effective methods.

The aim of this work is to determine the influence of the duration of synthesis, amplitude, and pulse duration of ultrasound-assisted synthesis on the final phase ratio and crystallite size of ZrO_2 nanoparticles.

Synthesis:

$ZrO(NO_3)_2 \cdot xH_2O$ and ethylenediamine were used in aqueous media

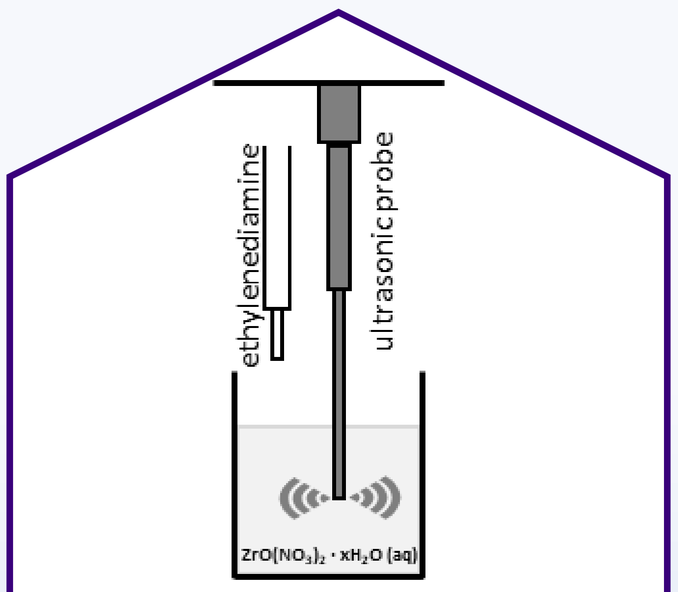

Fig. 1. Scheme of the preparation of zirconium dioxide nanoparticles

RESULTS AND DISCUSSION

FT-IR analysis of precursors doesn't show the presence of hydroxide OH group and refers to the synthesis of hydrated oxides.

After calcination peak at 504 cm^{-1} is present (Zr – O stretching). FT-IR analysis shows the same qualitative composition of precursors (and calcinated samples) so it doesn't depend on synthesis parameters.

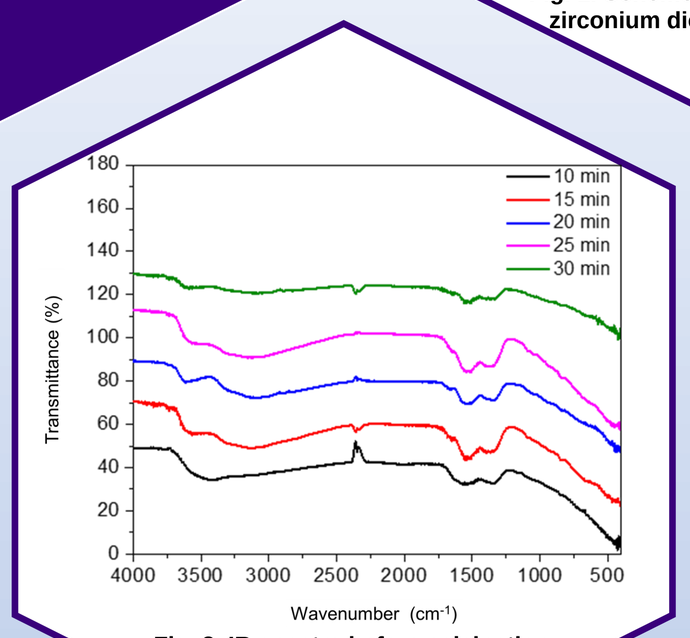

Fig. 2. IR spectra before calcination

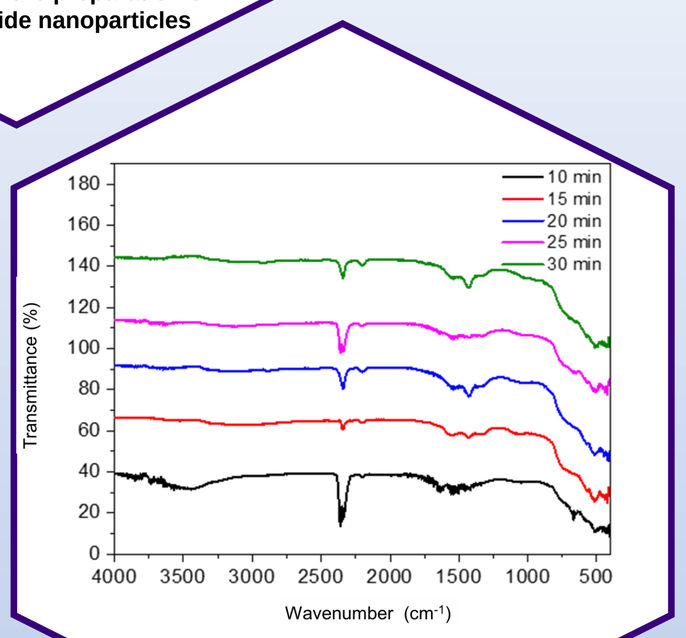

Fig. 3. IR spectra after calcination

Quantitative PXRD analysis show presence of monoclinic and tetragonal ZrO_2 phases in samples after calcination. Increased share of monoclinic phase was detected in samples sonicated up to 15 min and samples sonicated with ultrasound pulse of 1 – 2 s. Pure tetragonal phase is present only in a sample sonicated for 30 min.

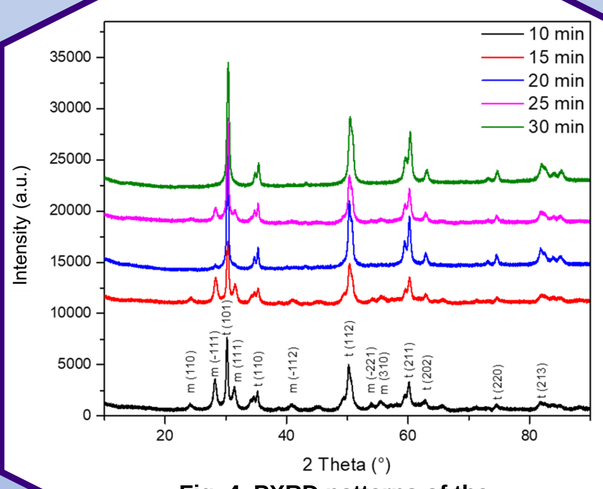

Fig. 4. PXRD patterns of the samples synthesized with different synthesis times

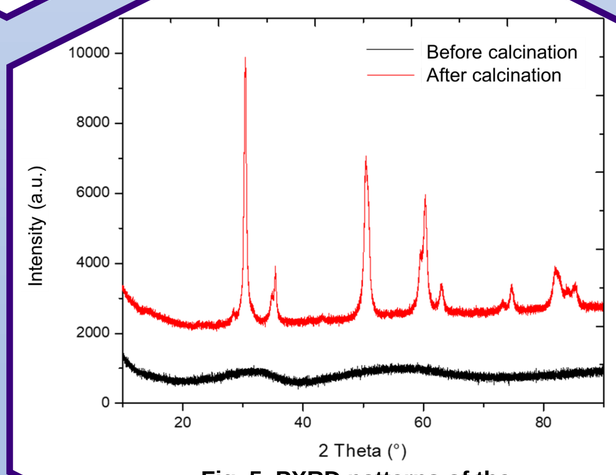

Fig. 5. PXRD patterns of the samples before and after calcination

CONCLUSIONS

The composition of the samples after calcination is mainly influenced by synthesis and pulse duration. Samples sonicated for longer than 15 min contain more than 90% of the tetragonal phase. Applying an ultrasonic pulse of 1 and 2 s produces 37.9% and 21.7% of the monoclinic phase, while a longer pulse, again, gives more than 90% of the tetragonal phase.

REFERENCES

[1] J. Clark, D. Macquarrie, Handbook of Green Chemistry & Technology, Wiley – Blackwell, Hoboken, New Jersey, 2002.

[2] C. V. Reddy, I. N. Reddy, J. Shim, D. Kim, K. Yoo, Ceramics International, 44 (2018) 12329–12339.