Hydrothermally and mechanically induced transformations in the porous and crystal structure of zirconium dioxide

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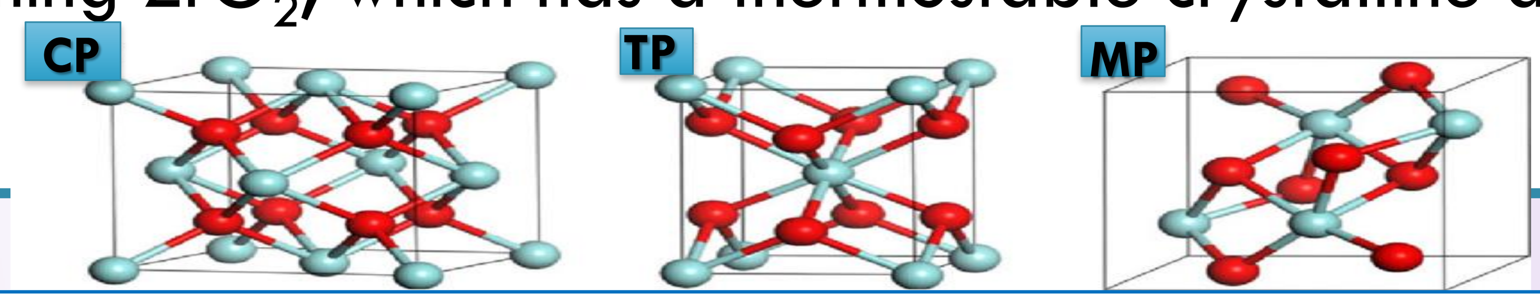
It is known that polymorphism is characteristic of zirconium dioxide: monoclinic (MP), cubic (CP) and tetragonal (TP) phases. ZrO_2 as supports for catalysts is used to 500 °C as a rule. The absence of phase transition and maximum resistance to sintering are the main requirements for support.

The purpose of this work is obtaining ZrO_2, which has a thermostable crystalline and porous structure.



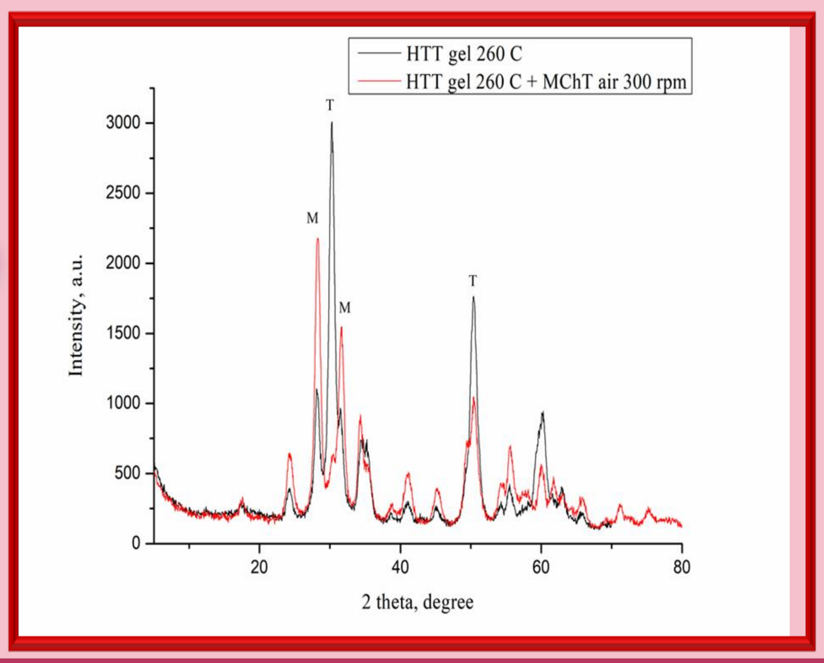
EXPERIMENTAL

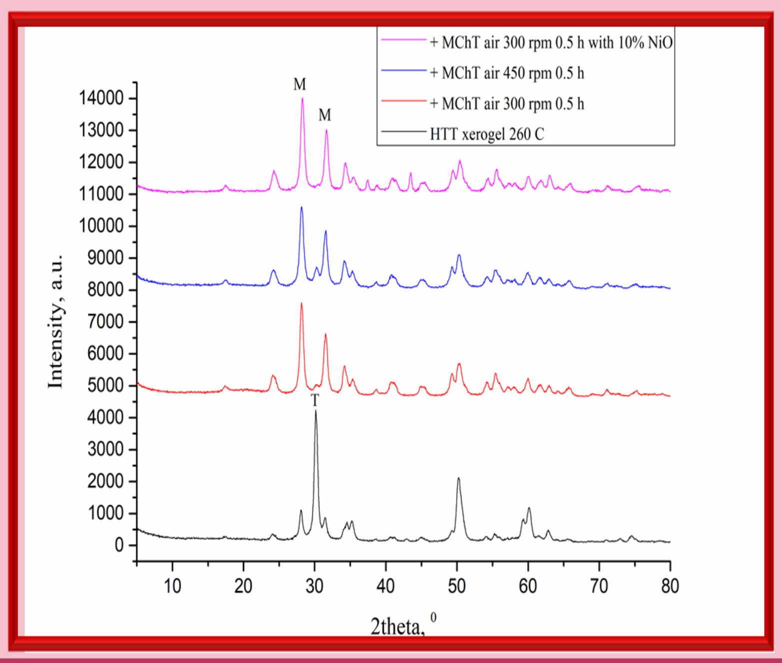
We have used successive hydrothermal (HTT) and mechanochemical (MChT) treatments to synthesize a stable monoclinic modification and create a mesoporous structure that is more resistant to sintering processes.

R E S U L T S

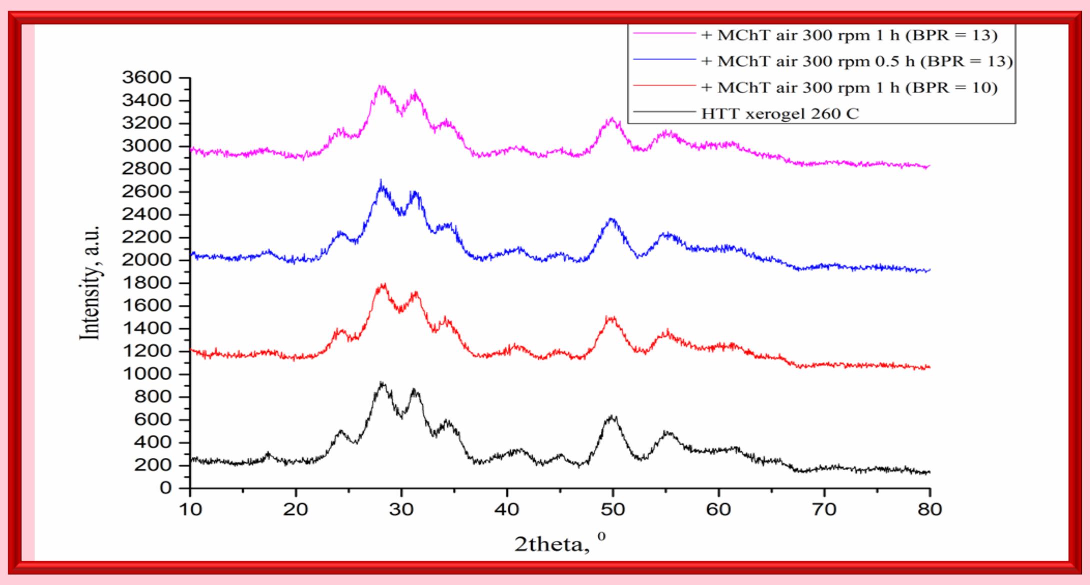
pH 9: HTT foms mixture of MP with low temperature TP and next mild dry MChT leads to formation almost pure MP

This is an unexpected previously undescribed result.

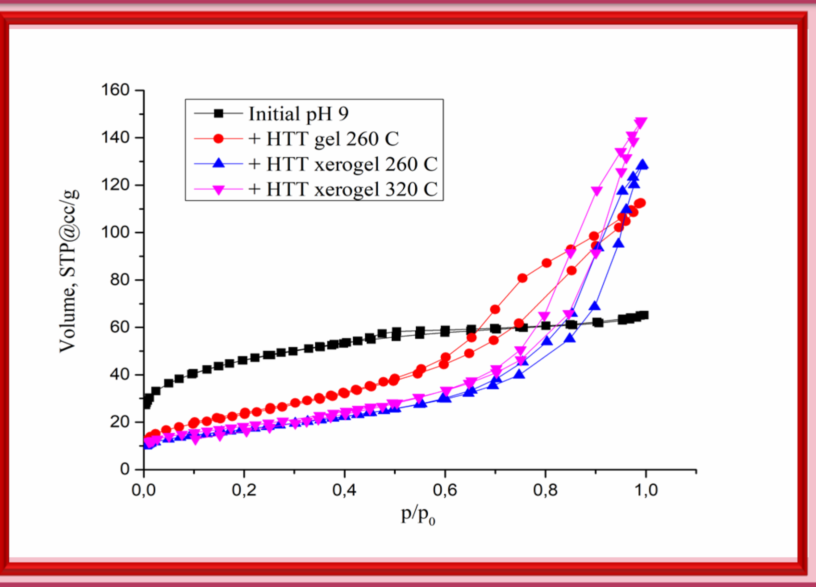


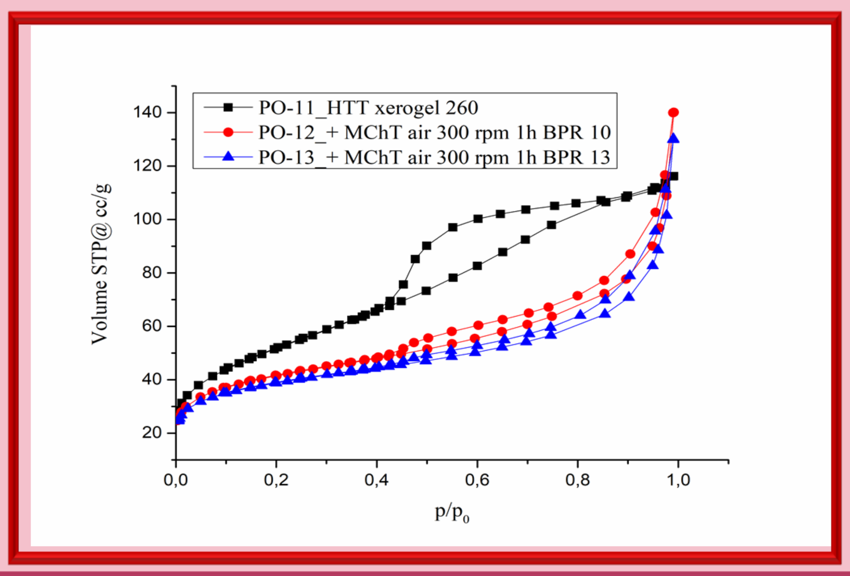


pH 7: HTT of wet gel at 300 °C for 5 h leads to formation of pure high-crystalline MP and phase composition does not change after next MChT



HTT forms developed mesoporous structure that is slightly changed due to next MChT in both cases





N	Conditions of modification	S, m^2/g	V_{me}, cm^3/g	d_{me}, nm	MP Content, %
PO-1	Initial pH 9	166	0.06	3,5	-
PO-2	+ HTT xerogel 260 C	61	0.20	7.9	32
PO-3	HTT xerogel 260 C + MChT 300 rpm 0.5h BPR 10	66	0.16	6.5	91
PO-4	HTT xerogel 260 C + MChT 450 rpm 0.5h BPR 10	67	0.15	3.9	85
PO-5	HTT xerogel 260 C + MChT 300 rpm 0.5h BPR 10 with 10% NiO	63	0.15	6.5	91
PO-6	HTT xerogel 320 C	66	0.23	9.5	46
PO-7	+ MChT 300 rpm 0.5h BPR 10	74	0.19	6.5	88
PO-8	HTT gel 260 C	87	0.175	6.6	40
PO-9	+ MChT 300 rpm 0.5h BPR 10	100	0.165	3.7	85

N	Conditions of modification	S, m^2/g	V_{me}, cm^3/g	d_{me}, nm	MP Content, %
PO-10	Initial pH 7 xerogel	130	0.07	2.6	-
PO-11	+ HTT xerogel 260 C	184	0.18	3.7	95
PO-12	HTT xerogel 260 C + MChT 300 rpm 1h BPR 10	149	0.215	3.7	95
PO-13	HTT xerogel 260 C + MChT 300 rpm 1h BPR 13	139	0.20	3.7	95
PO-15	+ HTT gel 300 C dispersion	60	0.17	9.7	100
PO-15-1	HTT gel 300 C + drying 150 C	60	0.17	9.7	100
PO-16	HTT gel 320 C 1h + TT 500 C	61	0.24	9.7	95
PO-17	Initial + TT 500 C	27	0.07	6.5	88