

High-temperature-treated LTX zeolites as heterogeneous catalysts for the Hock cleavage

Jan Dröner¹, Karim Bijerch¹, Peter Hausoul², Regina Palkovits², and Matthias Eisenacher^{1,*}

- ¹ Circular Transformation Lab Cologne, TH Köln-University of Applied Sciences, 51379 Leverkusen, Germany
karim.bijerch@smail.th-koeln.de (K.B.) matthias.eisenacher@th-koeln.de (M.E.)
- ² Institut für Technische und Makromolekulare Chemie, RWTH Aachen University, 52074 Aachen, Germany

* Correspondence: matthias.eisenacher@th-koeln.de

Gas chromatography

Cumene hydroperoxide disproportionates due to the high temperatures during GC analyses. Because of this, triphenylphosphine was added to the samples for the GC samples to convert remaining cumene hydroperoxide to 2-Phenyl-propanol. The conversion was determined by the remaining amount of 2-Phenyl-propanol, yield and selectivity in relation to the detected phenol.

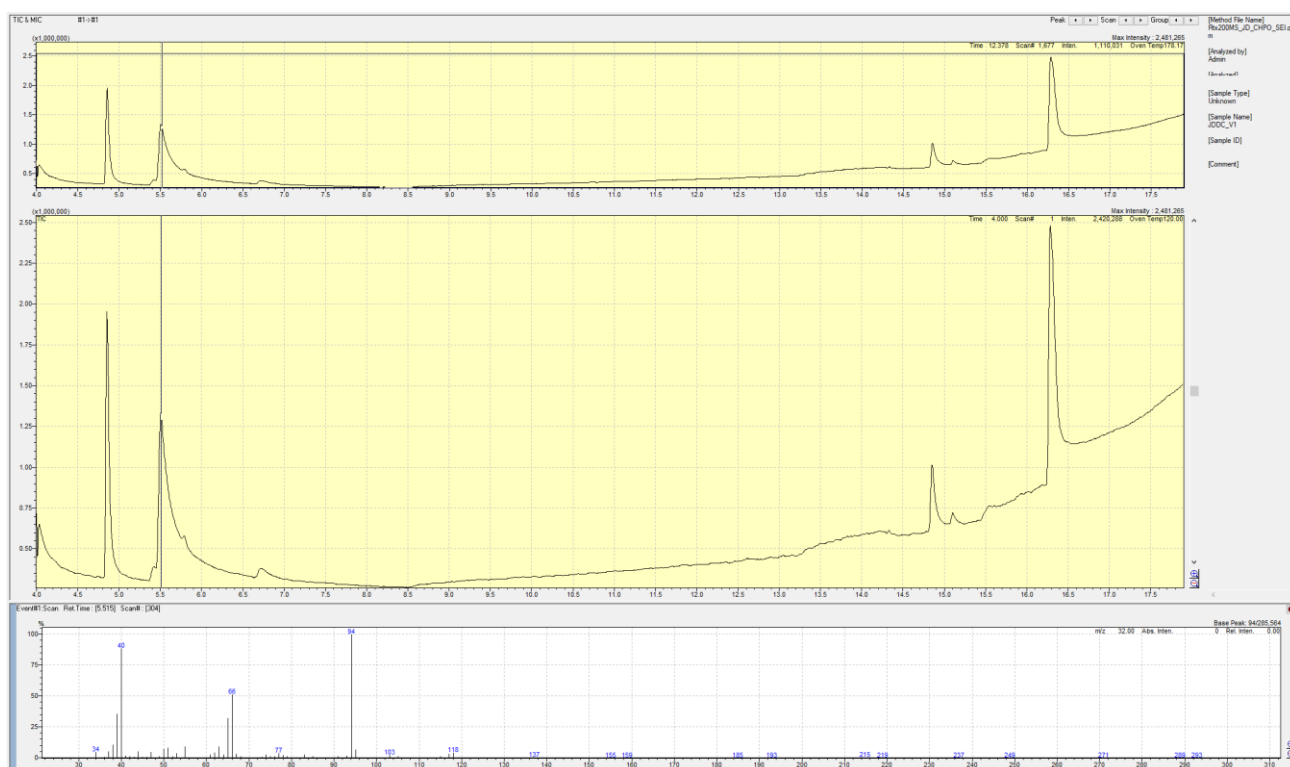


Figure S1. Exemplary chromatogram and spectrum of the product mixture of one of the experiments. The spectrum shown is that of phenol.

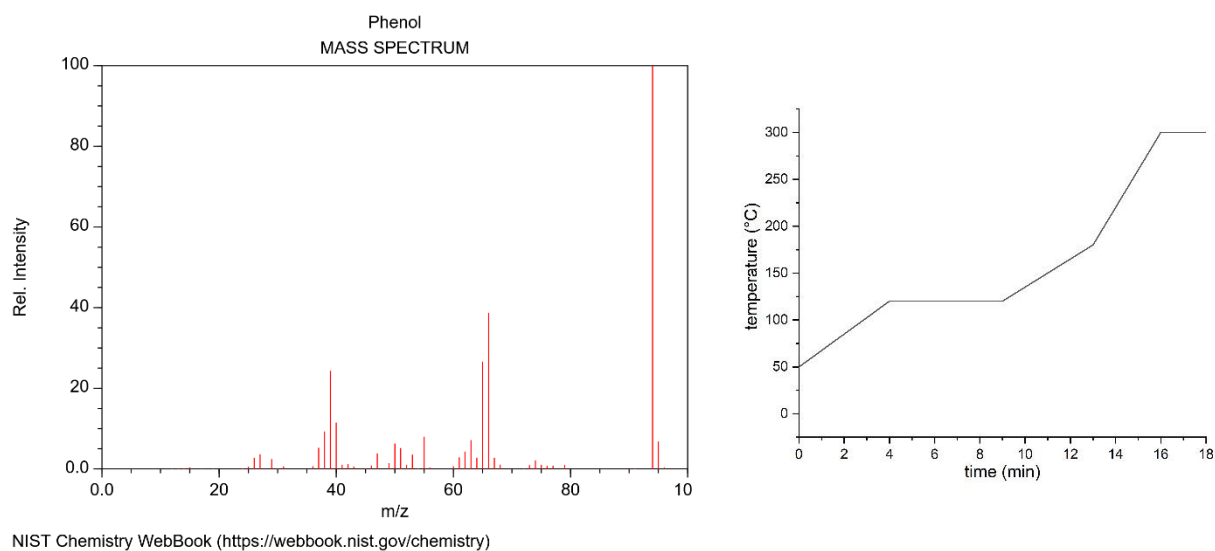


Figure S2. Left: Reference spectrum of phenol (<https://webbook.nist.gov/cgi/cbook.cgi?Spec=C108952&Index=0&Type=Mass&Large=on&SVG=on>, on 10.01.2023). Right: Temperature profile of the used GCMS-method.

BET analyses

Sample: LTXO

Operator:

Submitter:

File: I:\BET\FH köln\2021-1-122-LTXO.SMP

Started: 23.04.2021 08:34:55	Analysis adsorptive: N2
Completed: 23.04.2021 18:12:50	Analysis bath temp.: 77,300 K
Report time: 07.05.2021 04:11:09	Thermal correction: No
Sample mass: 0,2486 g	Warm free space: 15,7651 cm ³ Measured
Cold free space: 46,9637 cm ³	Equilibration interval: 5 s
Low pressure dose: None	Sample density: 1,000 g/cm ³
Automatic degas: No	

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 435,4328 m²/g

BET Surface Area: 417,1772 m²/g

t-Plot Micropore Area: 326,3933 m²/g

t-Plot external surface area: 90,7839 m²/g

BJH Adsorption cumulative surface area of pores

between 1,7000 nm and 300,0000 nm width: 71,0446 m²/g

BJH Desorption cumulative surface area of pores

between 1,7000 nm and 300,0000 nm width: 80,2135 m²/g

Pore Volume

t-Plot micropore volume: 0,170419 cm³/g

BJH Adsorption cumulative volume of pores

between 1,7000 nm and 300,0000 nm width: 0,087547 cm³/g

BJH Desorption cumulative volume of pores

between 1,7000 nm and 300,0000 nm width: 0,089518 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 4,9291 nm

BJH Desorption average pore width (4V/A): 4,4640 nm

DFT Pore Size

Volume in Pores	<	1,094 nm	:	0,00000 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,25817 cm ³ /g
Total Area in Pores	>=	1,094 nm	:	486,951 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,140286762$: 0,206806 cm³/g

Median pore width: 0,9183 nm

Figure S3. Report of BET analysis of a not calcined LTX zeolite.

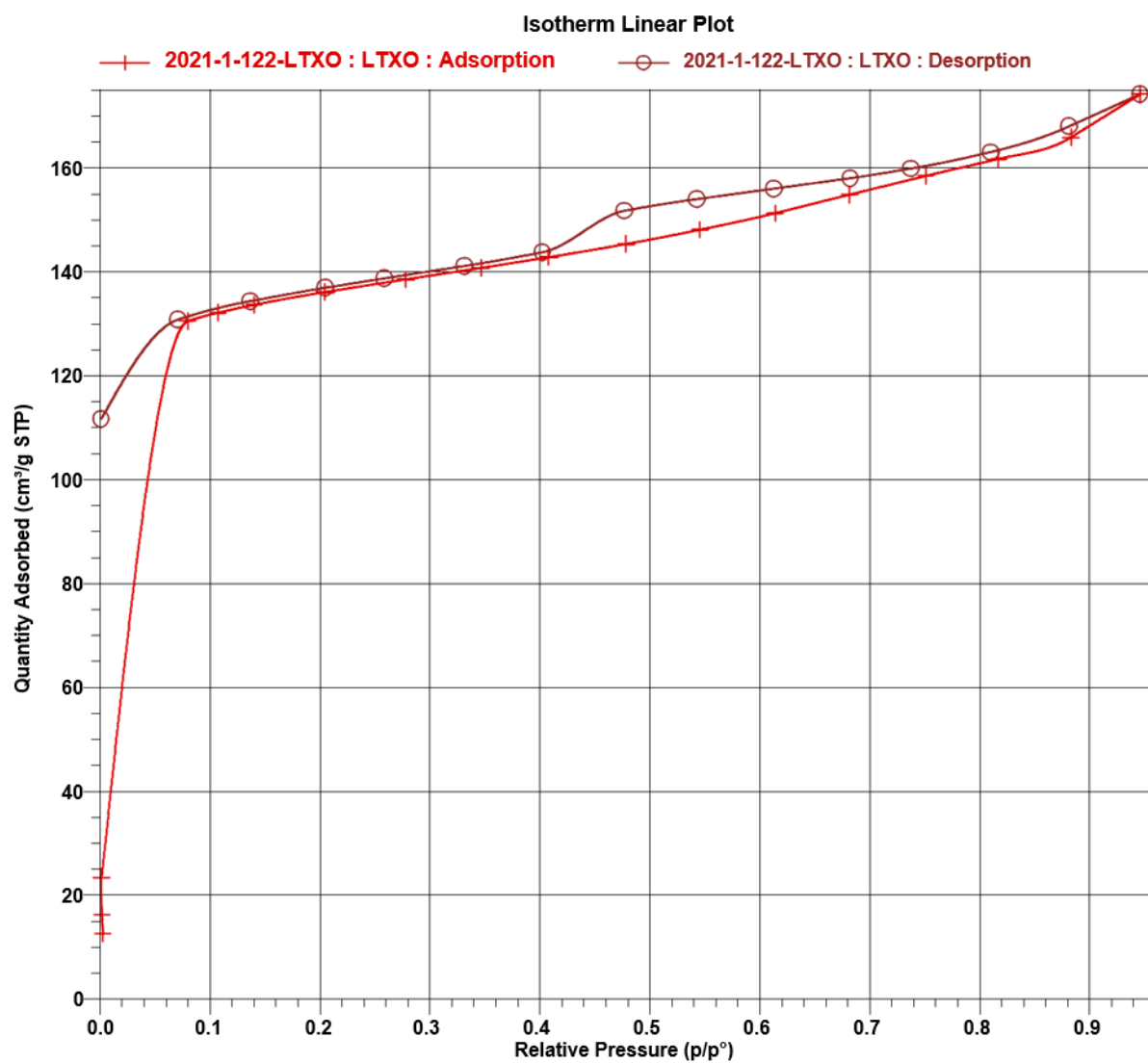


Figure S4. Linear plotted isotherm of a not calcined LTX zeolite.

Sample: LTXO-500
 Operator:
 Submitter:
 File: I:\BET\FH köln\2021-1-220-LTXO-500.SMP

Started: 23.04.2021 08:34:57	Analysis adsorptive: N2
Completed: 23.04.2021 17:05:30	Analysis bath temp.: 77,300 K
Report time: 07.05.2021 04:06:32	Thermal correction: No
Sample mass: 0,2116 g	Warm free space: 16,2072 cm ³ Measured
Cold free space: 48,2114 cm ³	Equilibration interval: 5 s
Low pressure dose: None	Sample density: 1,000 g/cm ³
Automatic degas: No	

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 304,3711 m²/g

BET Surface Area: 290,9394 m²/g

t-Plot Micropore Area: 213,6213 m²/g

t-Plot external surface area: 77,3180 m²/g

BJH Adsorption cumulative surface area of pores
 between 1,7000 nm and 300,0000 nm width: 67,1382 m²/g

BJH Desorption cumulative surface area of pores
 between 1,7000 nm and 300,0000 nm width: 80,2214 m²/g

Pore Volume

t-Plot micropore volume: 0,111946 cm³/g

BJH Adsorption cumulative volume of pores
 between 1,7000 nm and 300,0000 nm width: 0,097755 cm³/g

BJH Desorption cumulative volume of pores
 between 1,7000 nm and 300,0000 nm width: 0,101906 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 5,8241 nm

BJH Desorption average pore width (4V/A): 5,0812 nm

DFT Pore Size

Volume in Pores	<	0,522 nm	:	0,03303 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,21336 cm ³ /g
Total Area in Pores	>=	0,522 nm	:	297,117 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,138771842$: 0,142747 cm³/g

Median pore width: 0,8047 nm

Figure S5. Report of BET analysis of a LTX zeolite calcined at 500 °C.

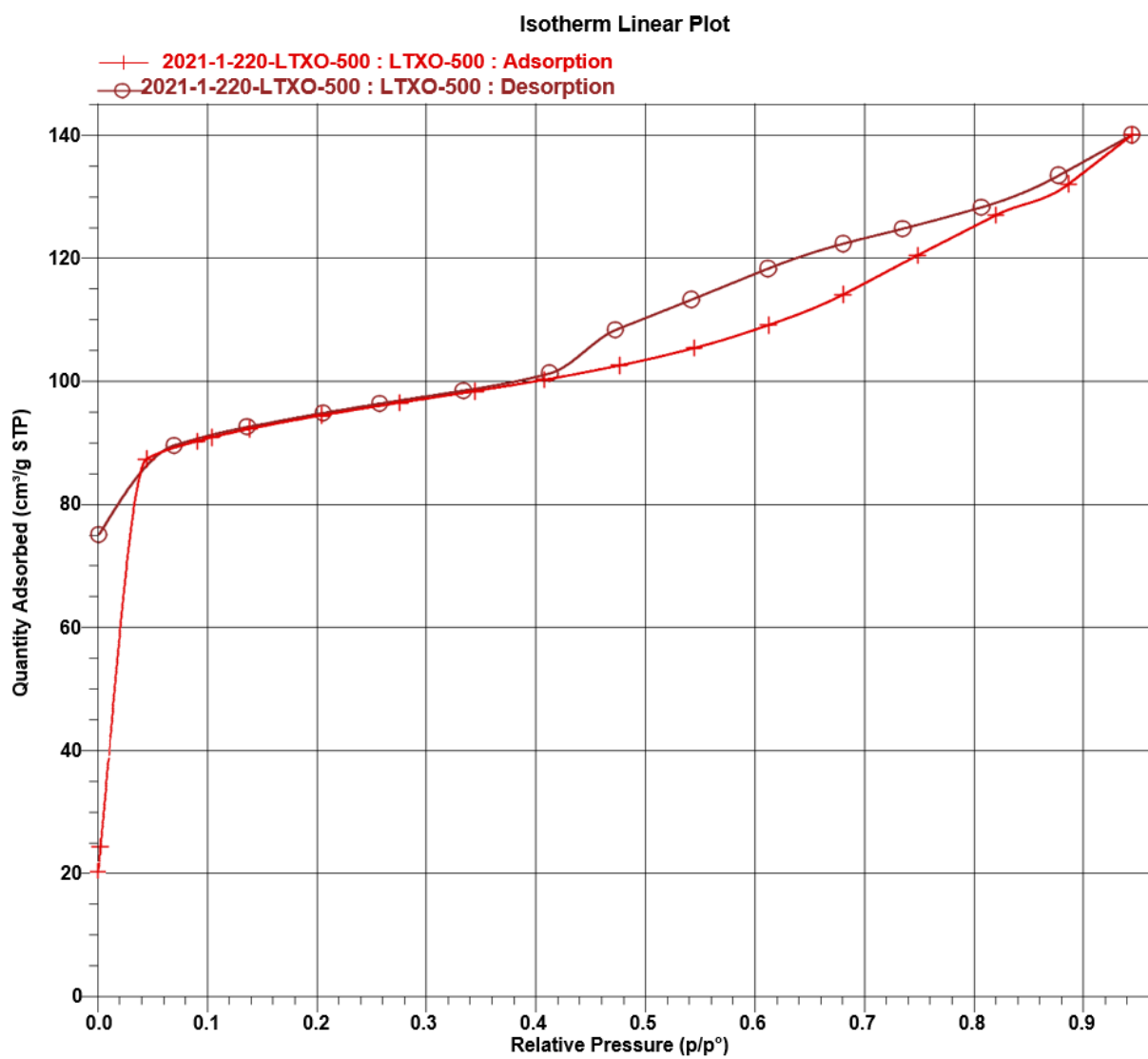


Figure S6. Linear plotted isotherm of a LTX zeolite calcined at 500 °C.

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 18,4416 m²/g

BET Surface Area: 18,8585 m²/g

t-Plot Micropore Area: 1,2182 m²/g

t-Plot external surface area: 17,6403 m²/g

BJH Adsorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 15,8302 m²/g

BJH Desorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 17,4152 m²/g

Pore Volume

t-Plot micropore volume: 0,000482 cm³/g

BJH Adsorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,031162 cm³/g

BJH Desorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,031733 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 7,8741 nm

BJH Desorption average pore width (4V/A): 7,2886 nm

DFT Pore Size

Volume in Pores	<	1,022 nm	:	0,00401 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,03219 cm ³ /g
Total Area in Pores	>=	1,022 nm	:	13,050 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,136465524$: 0,007442 cm³/g

Median pore width: 0,5822 nm

Figure S7. Report of BET analysis of a LTX zeolite calcined at 600 °C.

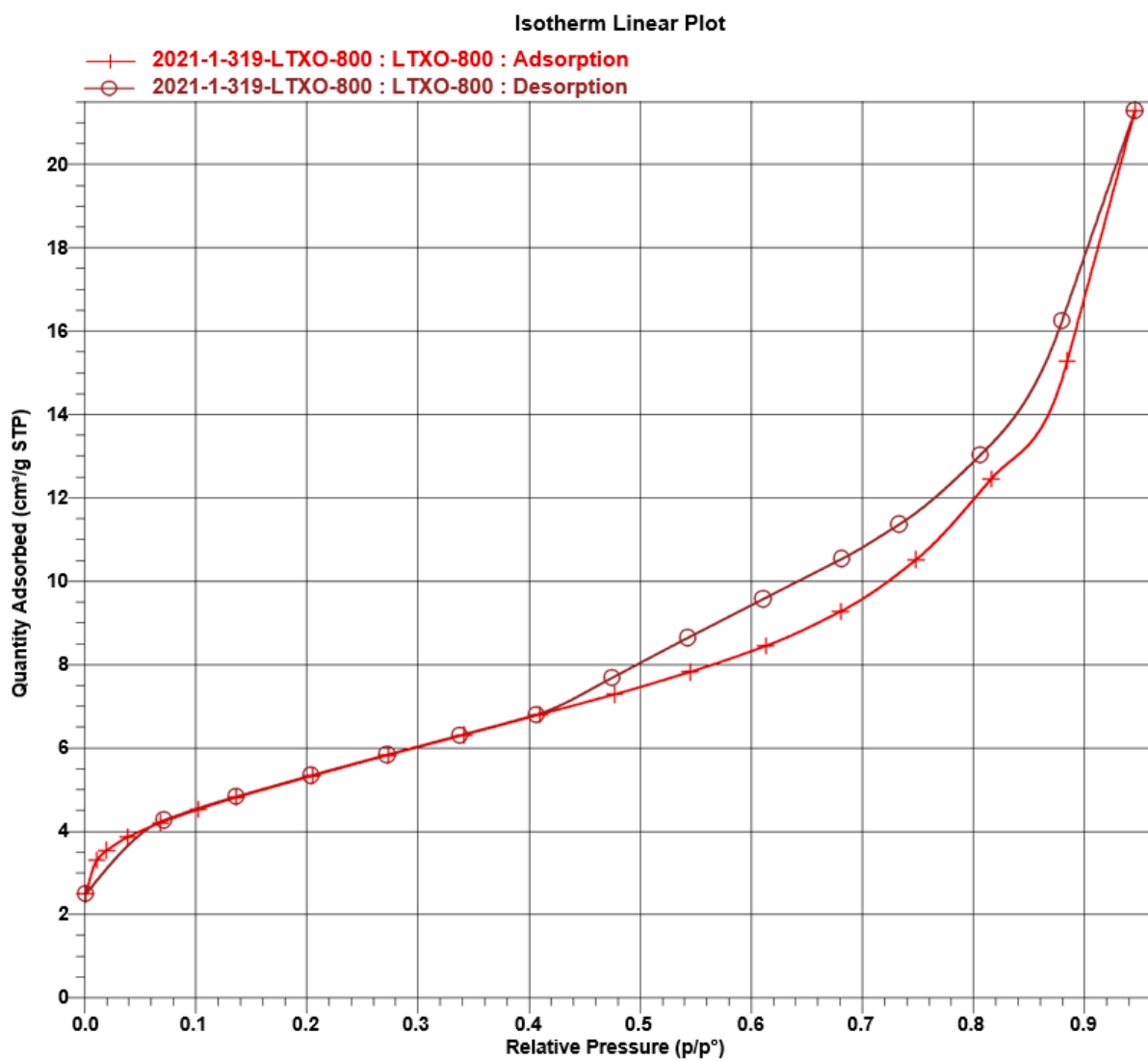


Figure S8. Linear plotted isotherm of a LTX zeolite calcined at 600 °C.

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 7,2239 m²/g

BET Surface Area: 7,4114 m²/g

t-Plot Micropore Area: 0,3310 m²/g

t-Plot external surface area: 7,0804 m²/g

BJH Adsorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 5,8571 m²/g

BJH Desorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 6,0560 m²/g

Pore Volume

t-Plot micropore volume: 0,000107 cm³/g

BJH Adsorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,009001 cm³/g

BJH Desorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,009020 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 6,1471 nm

BJH Desorption average pore width (4V/A): 5,9578 nm

DFT Pore Size

Volume in Pores	<	1,022 nm	:	0,00158 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,00957 cm ³ /g
Total Area in Pores	>=	1,022 nm	:	4,727 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,136543547$: 0,002901 cm³/g

Median pore width: 0,5835 nm

Figure S9. Report of BET analysis of a LTX zeolite calcined at 700 °C.

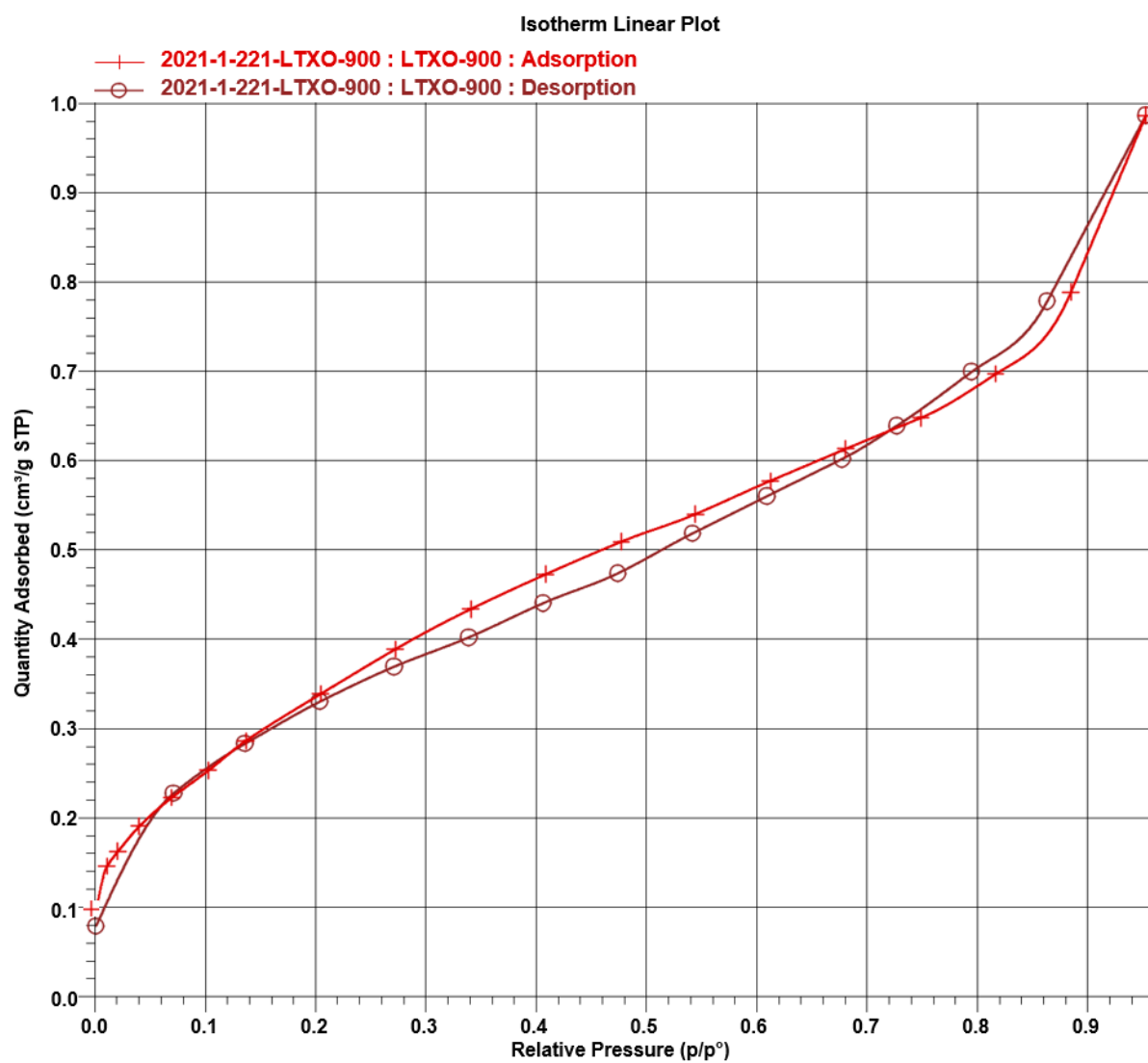


Figure S10. Linear plotted isotherm of a LTX zeolite calcined at 700 °C.

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 1,2321 m²/g

BET Surface Area: 1,3619 m²/g

t-Plot external surface area: 1,8389 m²/g

BJH Adsorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 1,3222 m²/g

BJH Desorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 1,2047 m²/g

Pore Volume

t-Plot micropore volume: -0,000286 cm³/g

BJH Adsorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,001514 cm³/g

BJH Desorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,001503 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 4,5807 nm

BJH Desorption average pore width (4V/A): 4,9921 nm

DFT Pore Size

Volume in Pores	<	1,022 nm	:	0,00014 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,00145 cm ³ /g
Total Area in Pores	>=	1,022 nm	:	0,898 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,136642039$: 0,000443 cm³/g

Median pore width: 0,7706 nm

Figure S11. Report of BET analysis of a LTX zeolite calcined at 800 °C.

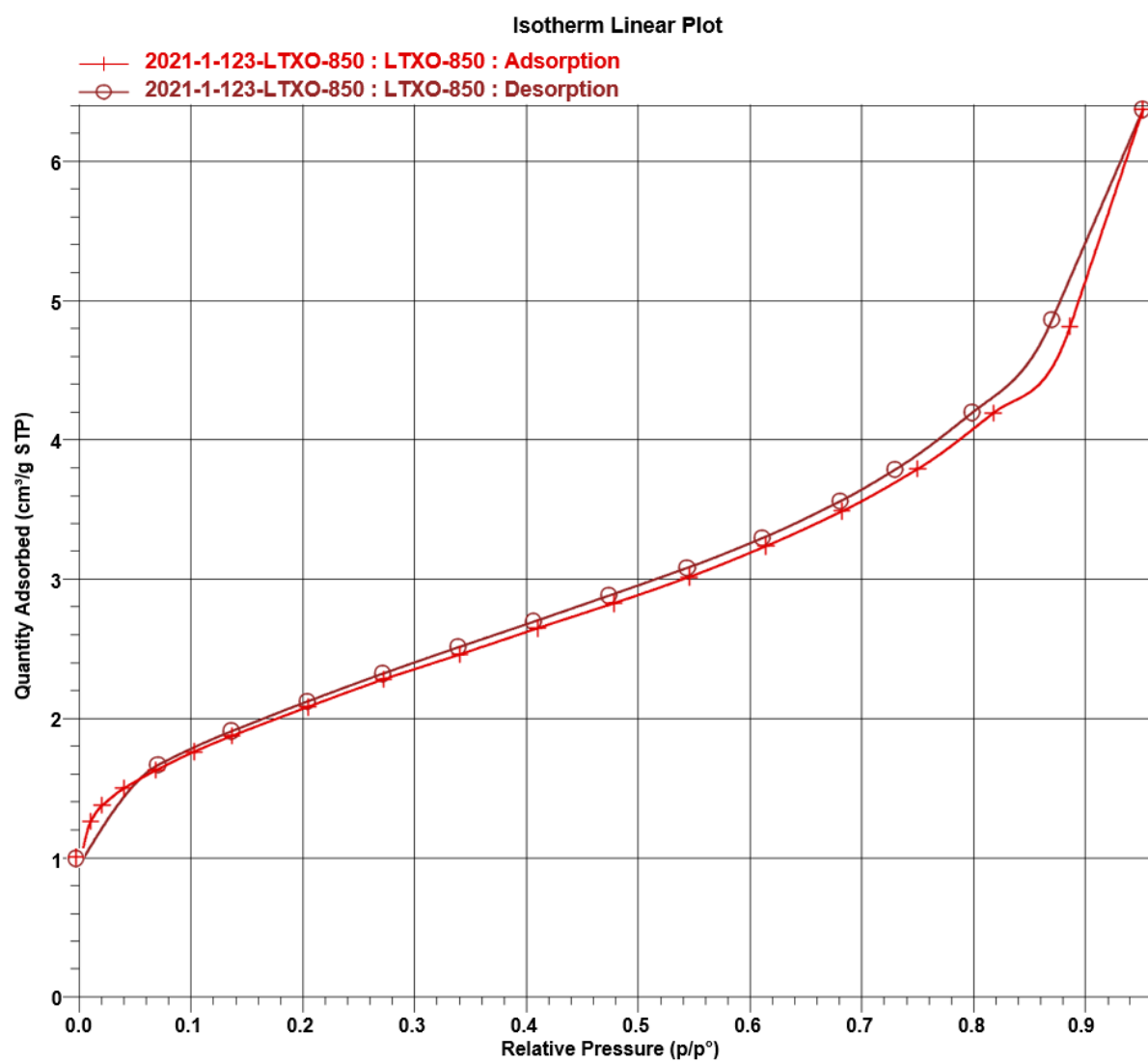


Figure S12. Linear plotted isotherm of a LTX zeolite calcined at 800 °C.

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 0,4770 m²/g

BET Surface Area: 1,2982 m²/g

t-Plot external surface area: 1,3375 m²/g

BJH Adsorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 0,4521 m²/g

BJH Desorption cumulative surface area of pores
between 1,7000 nm and 300,0000 nm width: 0,5885 m²/g

Pore Volume

t-Plot micropore volume: -0,000409 cm³/g

BJH Adsorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,000527 cm³/g

BJH Desorption cumulative volume of pores
between 1,7000 nm and 300,0000 nm width: 0,000608 cm³/g

Pore Size

BJH Adsorption average pore width (4V/A): 4,6655 nm

BJH Desorption average pore width (4V/A): 4,1302 nm

DFT Pore Size

Volume in Pores	<	1,022 nm	:	0,00000 cm ³ /g
Total Volume in Pores	<=	44,883 nm	:	0,00065 cm ³ /g
Total Area in Pores	>=	1,022 nm	:	0,464 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,136748000$: 0,000119 cm³/g

Median pore width: 1,2002 nm

Figure S13. Figure 7: Report of BET analysis of a LTX zeolite calcined at 900 °C.

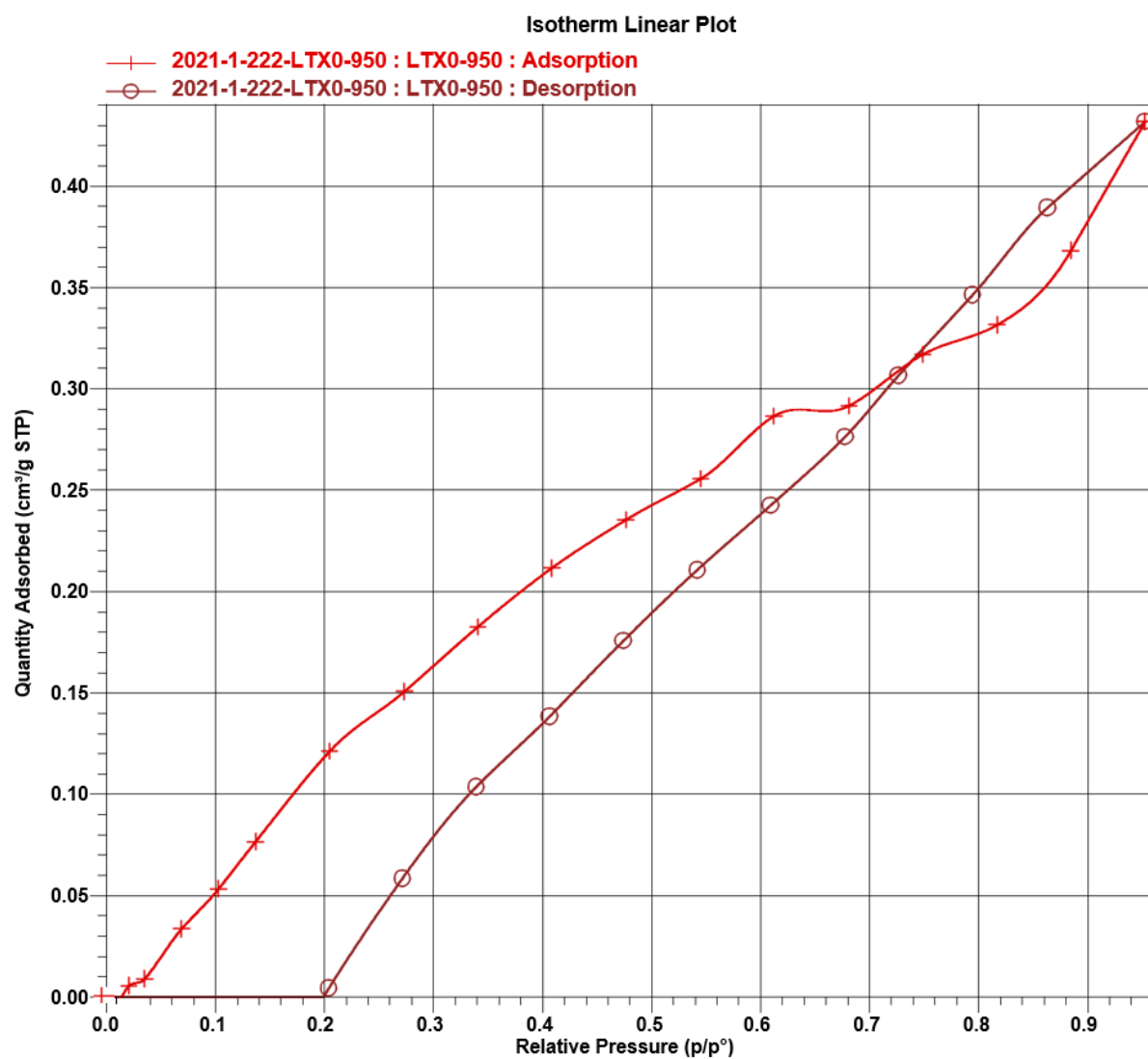


Figure S14. Linear plotted isotherm of a LTX zeolite calcined at 900 °C.

Summary Report

Surface Area

Single point surface area at $p/p^\circ = 0,300000000$: 0,0547 m²/g

BET Surface Area: 0,0513 m²/g

t-Plot Micropore Area: 0,2659 m²/g

t-Plot external surface area: -0,2145 m²/g

Pore Volume

t-Plot micropore volume: 0,000133 cm³/g

DFT Pore Size

Volume in Pores	<	1,022 nm	:	0,00001 cm ³ /g
Total Volume in Pores	<=	3,811 nm	:	0,00004 cm ³ /g
Area in Pores	>	3,811 nm	:	0,000 m ² /g
Total Area in Pores	>=	1,022 nm	:	0,087 m ² /g

Horvath-Kawazoe

Maximum pore volume at $p/p^\circ = 0,102674837$: 0,000052 cm³/g

Median pore width: 0,8299 nm

Figure S15. Report of BET analysis of a LTX zeolite calcined at 1000 °C.

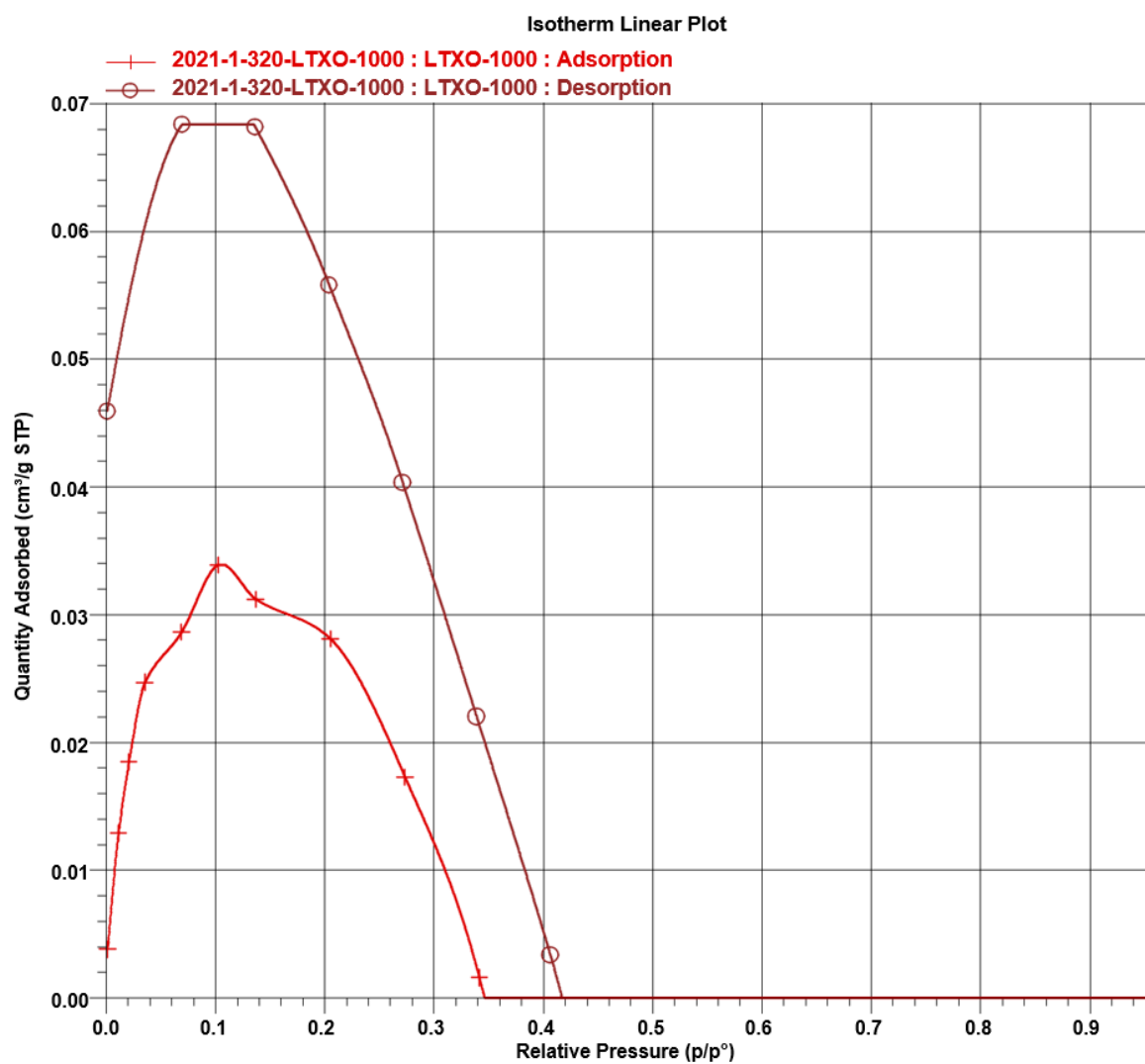


Figure S16. Linear plotted isotherm of a LTX zeolite calcined at 1000 °C.