

ALPHA Flux Test



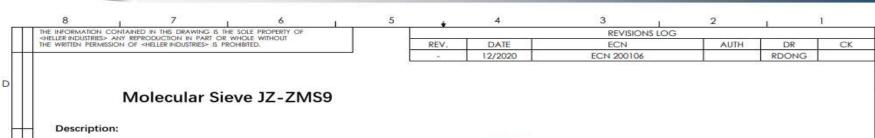


Zeolites BEFORE & AFTER Flux Test





X13 Zeolite



more than 9 angstroms. Applications:

1. Purification of gas in air separation plant, removal of H2O, CO2 and hydrocarbons.

JZ-ZMS9 is Sodium aluminosilicate, It could absorb the molecular which diameter is not

- 2. Dehydration and desulfurization (removal of H2S and mercaptan, etc.) of natural gas,LNG, liquid alkanes (propane, butane, etc.).
- Deep drying of general gases (e.g. compressed air, permanent gas).
- Drying and purification of synthetic ammonia.
- Desulfurization and deodorization of Aerosol.
- 6. CO2 removal from pyrolysis gas.

Specification:

Unit	sphere		cylinder	
mm	1.6-2.5	3-5	1/16*	1/8*
≥%	26.5	26.5	26	26
≥%	18	17.5	17.5	17.5
≥g/ml	0.64	0.62	0.62	0.62
≥N/Pc	25	80	25	65
≪₩	0.1	0.1	0.4	0.4
≤₩	1.5	1.5	2.0	2.0
	mm >% >% >% >% >N/Pc ≤%	mm 1.6-2.5 ≥% 26.5 ≥% 18 ≥g/ml 0.64 ≥N/Pc 25 ≤% 0.1	mm 1.6-2.5 3-5 ≥% 26.5 26.5 ≥% 18 17.5 ≥g/ml 0.64 0.62 ≥N/Pc 25 80 ≤% 0.1 0.1	mm 1.6-2.5 3-5 1/16* ≥% 26.5 26.5 26 ≥% 18 17.5 17.5 ≥g/ml 0.64 0.62 0.62 ≥N/Pc 25 80 25 ≤% 0.1 0.1 0.4

Standard Pakage:

sphere: 140kg/steel drum cylinder: 125kg/steel drum

OiuZhou Shanghai Jiuzhou Chemicals Co., Ltd

NOTE:

PROPERTIES: SPHERE DIAMETER: 3-5MM

Attention:

The product as desiccant cannot be exposed in the open air and should be stored in dry condition with air-proof package.

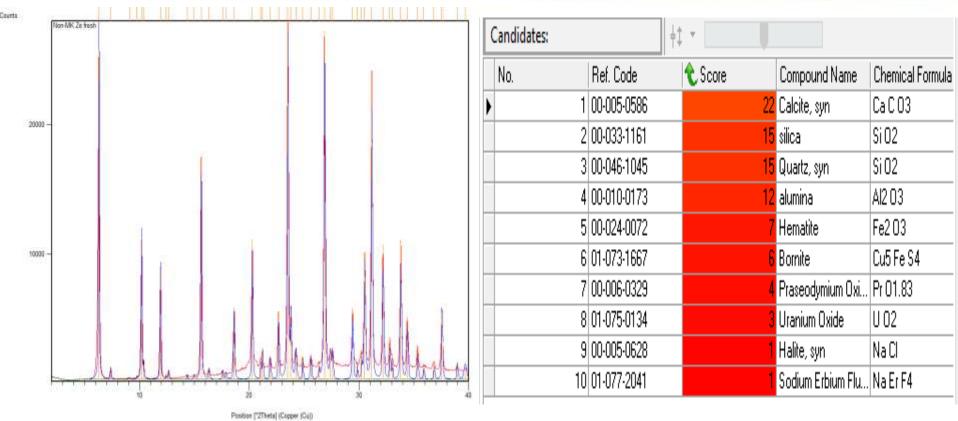
Address: Rm1111, No11, Yujinggang Rd, Jing'an District, Shanghai, China. Tel: +86 21 68769026 Fax: +86 21 68769036 E-mail: marketing@jiuzhouchemicals.com Website: www.jiuzhouchemicals.com

NO DEVIATIONS, SUBSTITUTIONS, OR EXCEPTIONS

	-		HEL	LER II	NDUS	TRIES INC	
			DIMENSIONS ARE IN MILLIMETERS UNLESS OTHERWISE SPECIFIED	MATERIAL SEE NOTES	JZ-ZMS	9 ZEOLITE, 13X GRADE	
THIRD ANGLE PROJECTION			TOLERANCES ARE: (ALL) DECIMAL PLACES: ±0.38 ANGULAR: ±1*	FINISH	DRAWN BY APPROVED BY	RDONG RAVIT	DATE 12/19/2
QUANTITY: SO. No:			BREAK ALL SHARP CORNERS (0.13 - 0.25) TOLERANCE ZONE PROJECTION ± 25.4 ALL MACHINED SURFACES 32	PROJECT/MFG No:	SIZE	2210186	revisi in
COMP. DATE:	NEXT ASSY	QTY	DO NOT SCALE DRAWING	SCALE	LAST SAVED BY	r: rpotineni 3/31/2021	SHEET 1 O



XRD - Fresh Zeolite - X13



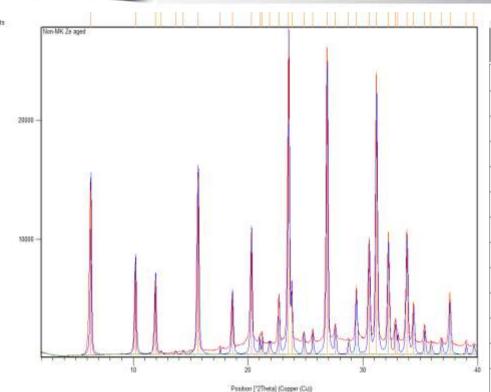
Zeolites are microporous, aluminosilicate minerals commonly used as commercial adsorbents and catalysts.

- Presence of Calcite is considered significant
- Zeolite extrudate size ~4mm
- Si -Al ratio ~3 to 1
- Pore size unknown





XRD - Aged Zeolite - X13 time in service - unknown



9	electe	d Candidate: 00-00	5-0586 †‡ 🕶		
	No.	Ref. Code	€ Score	Compound Name	Chemical Formula
١	1	00-005-0586	28	Calcite, syn	Ca C 03
	2	00-033-1161	17	silica	Si 02
	3	00-046-1045	17	Quartz, syn	Si 02
	4	00-010-0173	16	alumina	Al2 03
	5	00-024-0072	13	Hematite	Fe2 03
	6	00-024-1977	13	Sucrose	C12 H22 O11
	7	01-073-1667	7	Bornite	Cu5 Fe S4
	8	00-033-0664	7	burnt ochre	Fe2 03
	9	00-006-0329	4	Praseodymium Ox	ide 01.83
	10	01-075-0134		Uranium Oxide	U 02
	11	01-077-2041	1	Sodium Erbium Flu	Na Er F4
	12	00-005-0628	1	Halite, syn	Na Cl

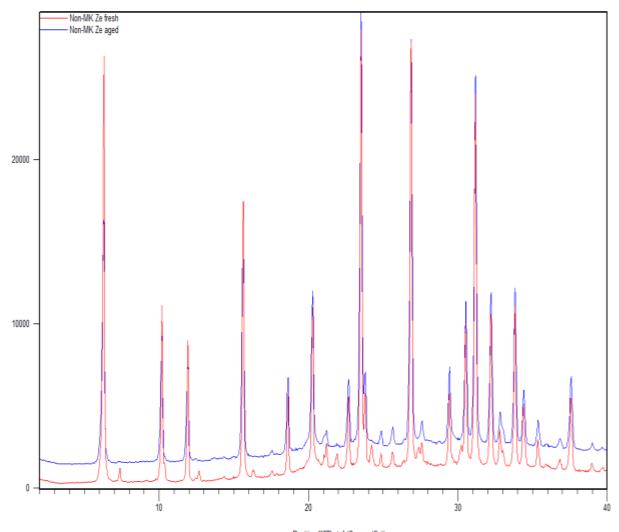
Sucrose, a disaccharide, (C12H22O11), from monomers. glucose and fructose likely originated in a condensation reaction from flux and thixotropic agents. Aged zeolite is 6.0mm in diameter, having adsorbed CO2 and water.

Burnt Ochre is a family of earth pigments, which includes yellow ochre, red ochre, purple ochre, sienna, and umber. The major ingredient of all the ochres is iron(III) oxide-hydroxide, known as limonite, which gives them a yellow color. Yellow ochre, FeO(OH)·nH 2O, is a hydrated iron hydroxide (limonite) also called gold ochre.





XRD comparative spectral overlap – X13 Zeolite – Fresh vs Aged

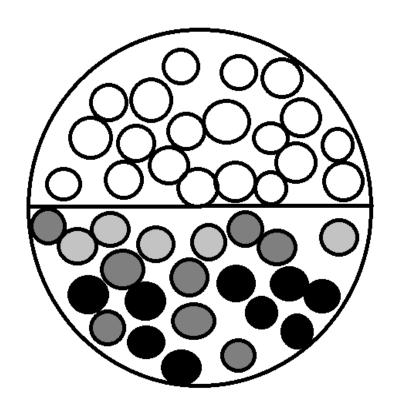


Aged and fresh samples with near identical structures, same zeolite

Structural features observed in the fresh sample, no longer evident in the aged sample



X13 Zeolite Bed Contaminant Profile



Mal-distribution of contaminants reportedly reside at the bottom region of the incoming zeolite bed

Suggesting non uniform flow and ineffective use of zeolite bed

Inlet flow straightener, eg, wire mesh may mitigate problem. Wire mesh matrix segments could improve zeolite life and performance

Irrespective of matrix design, inlet flow straightening may be necessary in a traditional monolith, metal, ceramic, extrudate





Flux composition

Rosin (resin) content is upward of 50-60% of a typical flux compound.

Abietic acid is the primary carboxylic acid in flux that serves to remove oxide formation from surfaces.

Rheological modifiers

Solvent – linear glycol

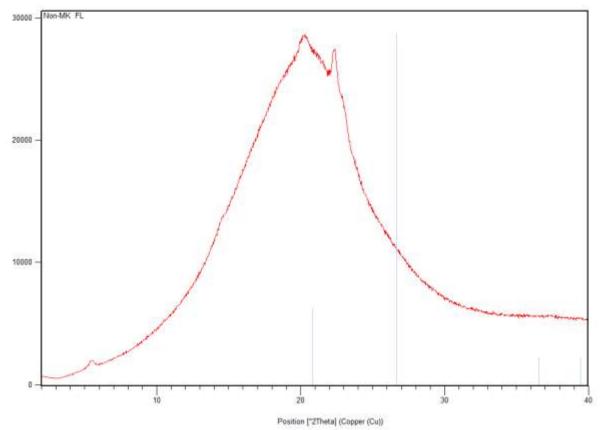
Small molecules

Pyrolysis or devolatilization of flux occurs at elevated temperatures in inert or (02) deprived environment.

In general, pyrolysis of organic substances produces volatile products and leaves a carbonrich solid residue. Pyrolysis, which leaves carbon compounds as the primary residue, carbonization. Pyrolysis is considered the first step in the processes of gasification or combustion.



XRD – Flux compound - Fresh



 No.
 Ref. Code
 Compound Name
 Chemical Formula

 ▶
 1 00-033-1161
 4 silica
 Si 02

 2 00-046-1045
 3 Quartz, syn
 Si 02

 3 00-005-0586
 2 Calcite, syn
 Ca C 03

Sample is largely amorphous suggesting high surface area and high surface energy

Highly viscous

Low crystallinity

Low or non-existent porosity

Flux composition:

- Small molecule
- Rheological Additives
- Abjetic acid
- Rosin/resin

CaCO3 and SiO2 binders Disaccharide formed in the furnace





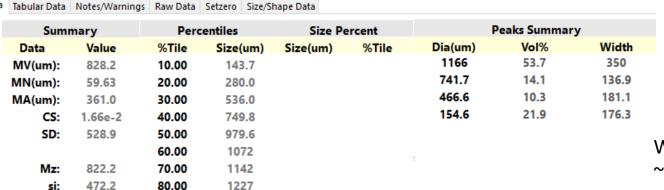
Ski:

Kg:

-0.37268

0.733

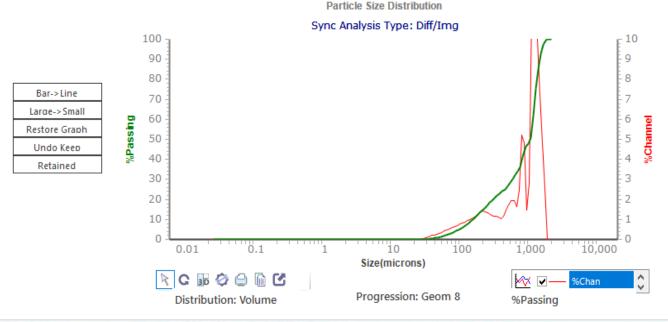
PSD-FLUX PSD by Microtrac FlowSync with nonpolar solvent Isopar-G Analyzed following 60s sonication



Wide PSD distribution ~144U to 1460U

Sample polymer-like mildly soluble in Isopar-G

Classification of Flux may improve deposition furnace contaminant profile



1358

1460

90.00

95.00

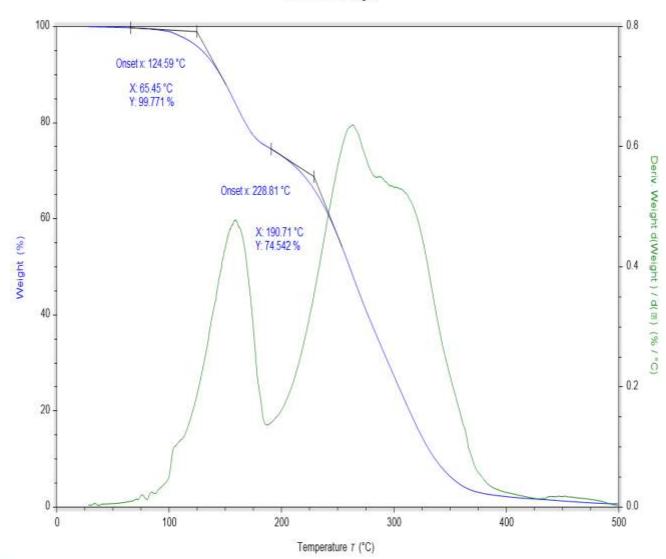




Flux

TGA/DTA in N2: Ramp 5.0c/min to 500c

Non-MK FL nitrogen

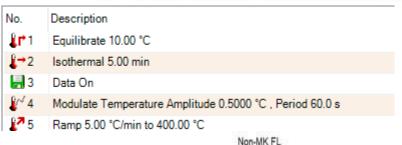


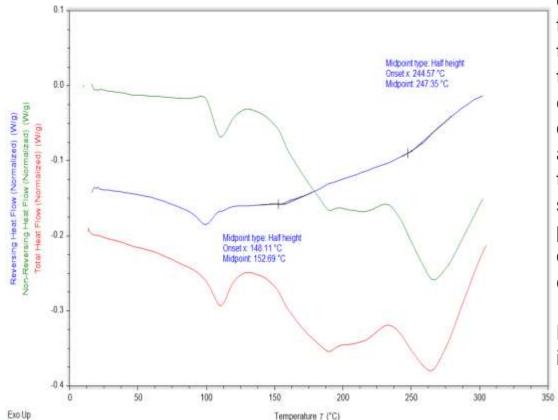
Onset ~65C – 500C

2 exotherms1 endotherm



Flux Characterized by DSC - Differential Scanning Calorimetry ~300C





DSC monitors changes in the heat effects associated with physical transitions and chemical reactions as a function of temperature. The DSC principle assumes that under constant pressure conditions the change of heat in any transition is equivalent to the change of enthalpy. The difference in heat flow to the sample and a reference is recorded as a function of temperature. DSC allows the user to study the kinetics of the physical processes and chemical reactions that occur during biodegradation, including enthalpy changes.

Not clear why heat in sample shows an increase with increasing T,



Characterization in Progress

SEM

flux – (particles, contaminants, features) aged zeolite - (pore size, contaminants)

TGA/air

flux - (how air and flow influence oxidation of HC's)

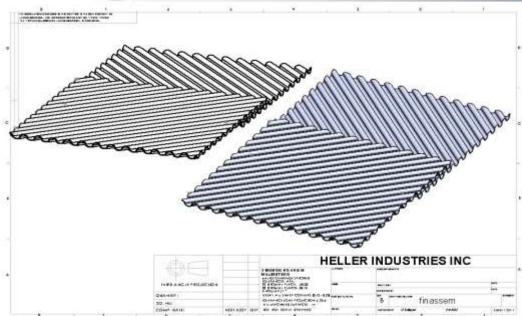
Porosity (N)

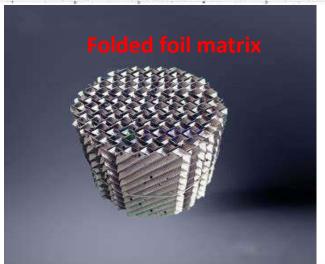
SA and micropore measurement Comparative, fresh and aged zeolite





H1 Cross Flow Metal Substrate Development – High Mass Transfer





Material: fecralloy-OCr21A16

Gauge: 50 micron

Corrugation height: 0.98mm Channel angle: 45 degree

Foil width: 106.30mm

Finished diameter: 138mmOD

5.433"D x 4.185"L

 $97.02'' = V \text{ (total cat V = } ^3.2L)$

No mantle

Nonionic wash

Foil roll with nodal matrix braze

Heat treat: 920C/2hrs/Air

Corrugation height 0.98mm

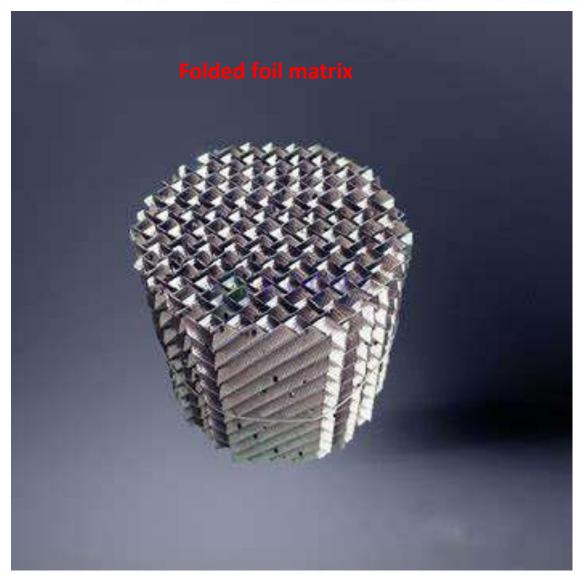
Corrugation width 2.1mm

4 wraps of flat foil on perimeter

Tolerance: +/- 0.38mmD Tolerance: +/- 0.13mmL



H1 Cross Flow Metal Substrate Development – High Mass Transfer Substrate



Material: fecralloy-OCr21A16

Gauge: 50 micron

Corrugation height: 0.98mm Channel angle: 45 degree

Foil width: 106.30mm

Finished diameter: 138mmOD

5.433"D x 4.185"L

 $97.02'' = V \text{ (total cat V = } ^3.2L)$

No mantle

Nonionic wash

Foil roll with nodal matrix braze

Heat treat: 920C/2hrs/Air

Corrugation height 0.98mm

Corrugation width 2.1mm

4 wraps of flat foil on perimeter

Tolerance: +/- 0.38mmD Tolerance: +/- 0.13mmL



Technology	Substrate	Туре	Dimensions	Quantity Requested	Quantity Received	Date Requested	Invoice Receive Date	PO
T			E CONT. A FIL	40		0 33.11	c in inon	000075464
Trap-baseline (no coating)	wire mesh		5.66D" x 0.5"L	10	Ť ·	-		P0337516/3
Trap-LPA (TH-100)	wire mesh	10 3000 F	5.66D" x 0.5"L	10				PO337516/3
H-Beta	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
H-Beta w/10% Cu	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
Cu-Y	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
ZSM-5	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
SCR	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
Cu-ZSM-5	metal monolith	straight channel	138mmD x 106.3mmL	3		6/2 & 7/15/22	NA	
DOC/DPF 5g/2:1:0	Cordierite	DOC/DPF	143.8*152/300	1				
DOC/GPF 5g/2:1:0	Cordierite	DOC/GPF	118.4*127/300	1		6/22/2022	P0375534	
CuSCR/GPF	Cordierite	CuSCR/GPF	118.4*127/300	1				
DOC/DPF 5g/2:1:0	Cordierite	DOC/DPF	143.8*152/300	1				
DOC/GPF 5g/2:1:0	Cordierite	DOC/GPF	118.4*127/300	1		7/15/2022	PC01153	
CuSCR/GPF	Cordierite	CuSCR/GPF	118.4*127/300	1		7. 7.		
H-Beta	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju	1	
H-Beta w/10% Cu	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju	I	
Cu-Y	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju	1	
ZSM-5	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju		
SCR	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju		
Cu-ZSM-5	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju	I	
DOC-DPF	Cordierite	floThru-300/400 cpsi	TBD	1		25-Ju		





Zeolyst-Zeolites: Test Candidates for Flux and Formic Applications

Source	Material	Pore SizeD	BET SA	Si-Al Ratio
Zeolyst	CBV-400	24.5A	730	5.1
Zeolyst	CBV-5524G	5.5A	425	50.1
Zeolyst	CBV-2314		425	23.1
Zeolyst	CBV-720	24.28A	780	30.1

Note:

- 2 Y zeolites and 2 ZSM-5 zeolites
- (CBV-2314, CBV-5524 are ZSM-5)
- (CBV-400, CBV-720 are Y Zeolites)
- CBV 5524G and CBV 2314 must be calcined at 500C for 1 hour in air to activate H form





Molecular Weight Distribution of Residue in Flux Reactor Cold Traps

GPC (Gel Permeation Chromatography) Eurofins

GPC separates organic compounds based on the size, (atomic radii), specifically by hydrodynamic volume

Size determination and separation are achieved through packed porous beads in a chromatographic column. Organics can be characterized by molecular wt and average molecular wt within a column.

Gels are used as the stationary phase for GPC. The pore size of a gel are carefully controlled in order to achieve a given separation. Other desirable properties of the gel forming agent are the absence of ionizing groups and, in each solvent, low affinity for the substances to be separated

The column used for GPC is filled with a microporous packing material. The column is filled with the gel.





XRF result: Wash-coat scrapped from commercial metal monolith

TEST REPORT

		试验结果	
		Test Result	
委托人	张元	Sample submission date	2022-06-20

	XRFUQ 数据					
Pat number	Sample description		Component	Test value %	Test method	
			Al2O3	65.593	3 5	
			CaO	0.120		
			CeO2	10.768		
			Cr2O3	0.342		
			Fe2O3	2.898		
220.5550	Scratching		La2O3	1.771	ECTCU II 000	
2205770	powder		MgO	0.052	ECTC/I-AL-080	
		1	MnO	1.881		
		1	Na2O	0.322		
			P2O5	0.600		
			SO3	0.652		
	/4		SiO2	14.203		

Precious metals deemed too low in concentration to resolve signal to noise interference in providing accurate measurement

Wash-coat loading deemed very low

Wash-coat reported to be very difficult to remove from substrate, suggesting low porosity, low wash coat loading

99.202% wash-coat materials, with PM & contaminant balance





ICP results suggest low PM and wash-coat content

		Dark, before reger	neration	Light, after regneration		
Component	mg/brick	mg/in³	g/in³	mg/brick	mg/in³	g/in³
Pd	28.58	0.325883694	0.000325884	30.451	0.347217788	0.000347218
Pt	29.32	0.334321551	0.000334322	22.838	0.26041049	0.00026041
Al	3288.1	37.49258837	0.037492588	4783.968	54.54923603	0.054549236
Ва	2.15	0.024515393	2.45154E-05	2.53	0.028848347	2.88483E-05
Ca	14.53	0.165678449	0.000165678	35.341	0.402976055	0.000402976
Cr	20.87	0.237970353	0.00023797	16.894	0.192633979	0.000192634
Fe	240.73	2.744925884	0.002744926	273.328	3.116624857	0.003116625
Mg	1.15	0.013112885	1.31129E-05	2.115	0.024116306	2.41163E-05
Mn	446.67	5.093158495	0.005093158	479.181	5.46386545	0.005463865
Ni	81.84	0.9331813	0.000933181	363.32	4.142759407	0.004142759
La	626.84	7.147548461	0.007147548	599.95	6.840935006	0.006840935
Ce	749.98	8.551653364	0.008551653	1020.632	11.63776511	0.011637765
			0.063064538			0.087007389





SEM - Scanning Electron Microscopy for Aged Zeolite

Bulk analysis - Morphology technique - back scattered electrons penetration at various depths Irregular Particle Morphology

Pore size, contamination profile not determined

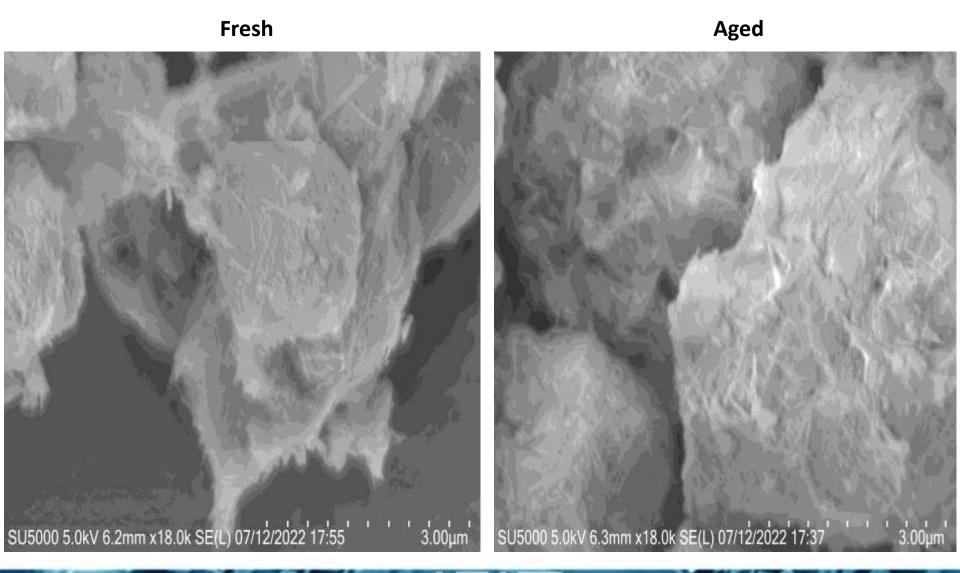
Fresh 90x

Aged 90x

SU5000 5.0kV 6.1mm x90 SE(L) 07/12/2022



SEM – X13 Zeolite 18KX

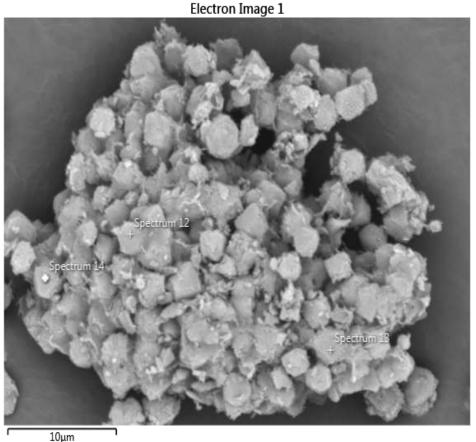


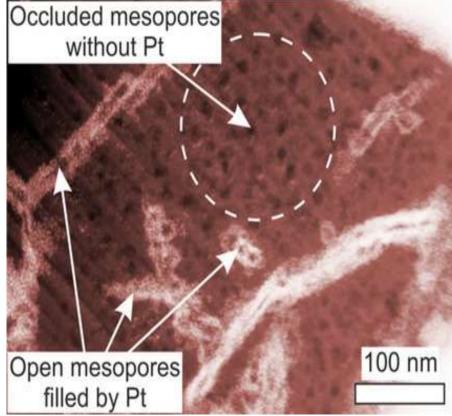


Primary zeolite particles ~ 1 microns D Zeolite pores ~ 1-10A D

H₃C-Pt-CH; +e beam → Pt tracking

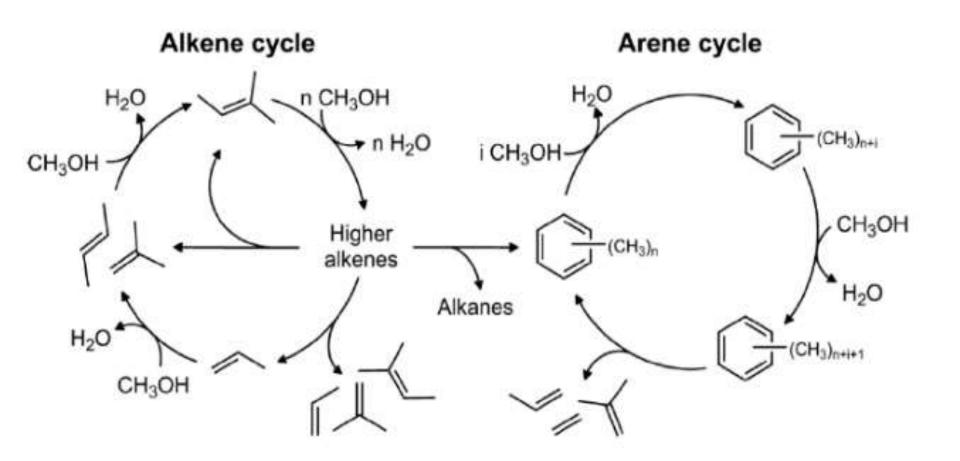
X13 3KX





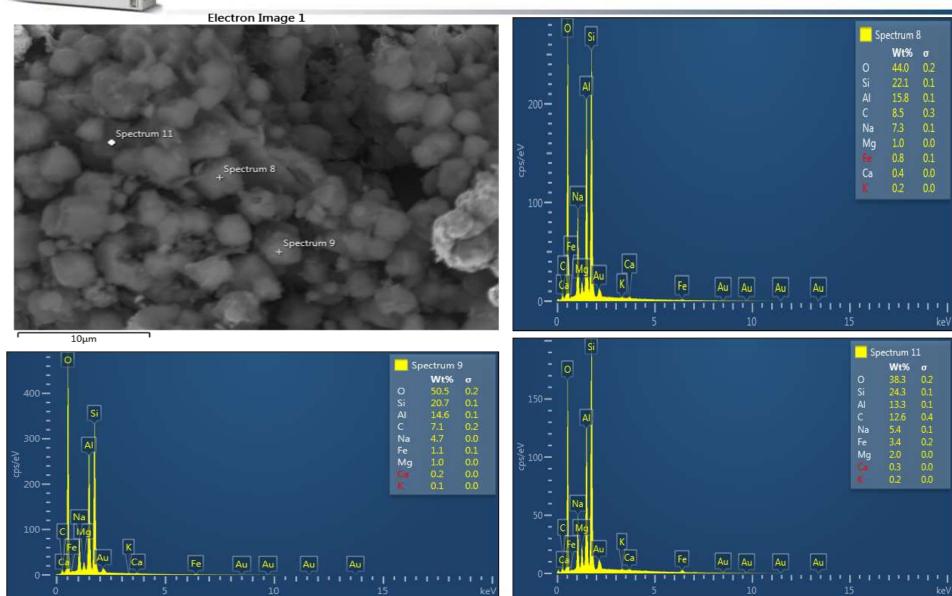


Zeolite HC Cracking Kinetics - Condensation (Exothermic) Rx

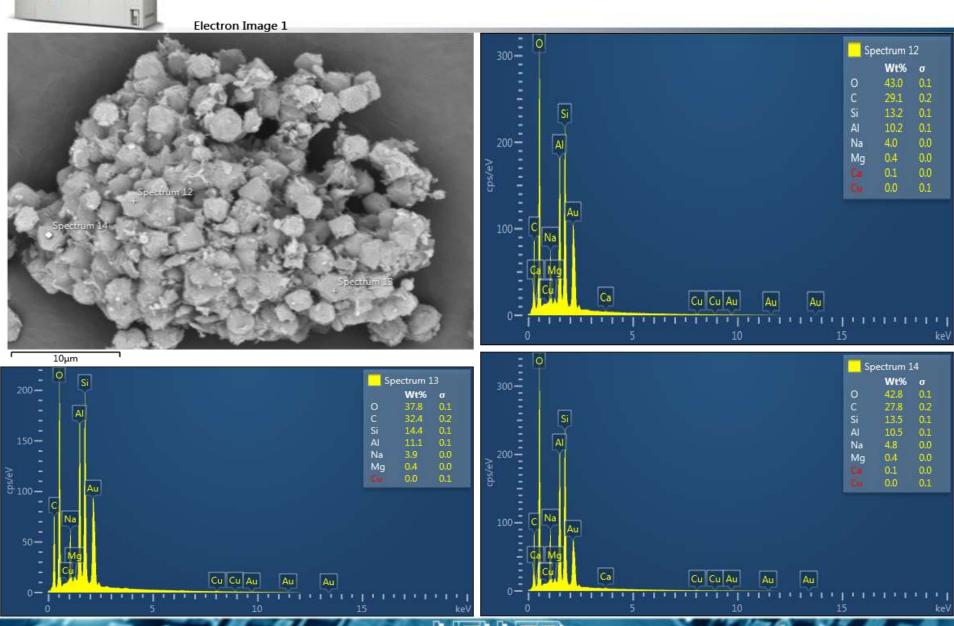


F

Aged Zeolite – SEM/EDX 3KX



Fresh Zeolite – SEM/EDX 3KX



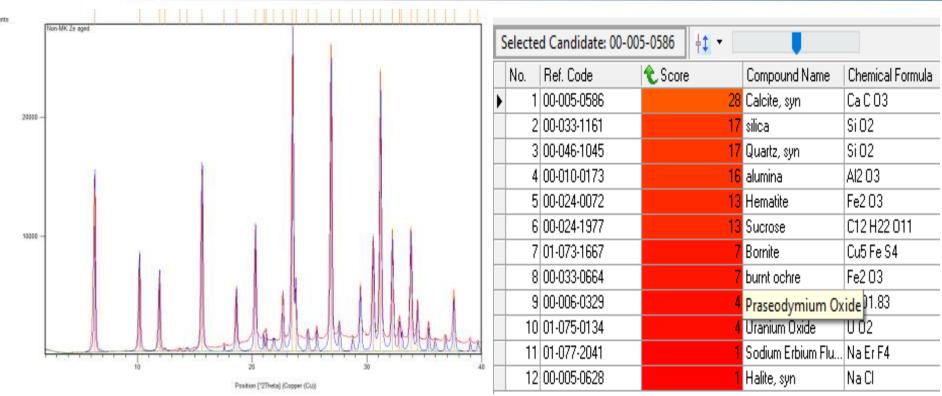


SEM – EDX Comparative Contaminant Summary - X13 Zeolite

		Zeolite – Fre	sh		Zeolite - Aged			
				average			;	average
0	43	37.8	42.8	average 41.20	40.4	45.3	44.2	43.30
C	29.1		27.8		22 7	18.1	17.6	22.80
Si	13.2		13.5		11 5	17	18	15.50
Al	10.2		10.5		7.0	12.5	12.8	11.07
Na	4		4.8			5.5	5.6	5.60
Mg	0.4		0.4			1	2	1.33
Ca	0.1		0.1			0.2	0.3	0.27
Cu	0.1		0.1			0.2	0	0.00
Fe	0		0			1.1	0.95	0.95
K	0	_	0			0.2	0.5	0.13
• •	_	/ · · · · · · · · · · · · · · · · · · ·		0.00	0.1	0.2	0.1	0.13



XRD - Aged Zeolite - X13 (time in service - unknown)



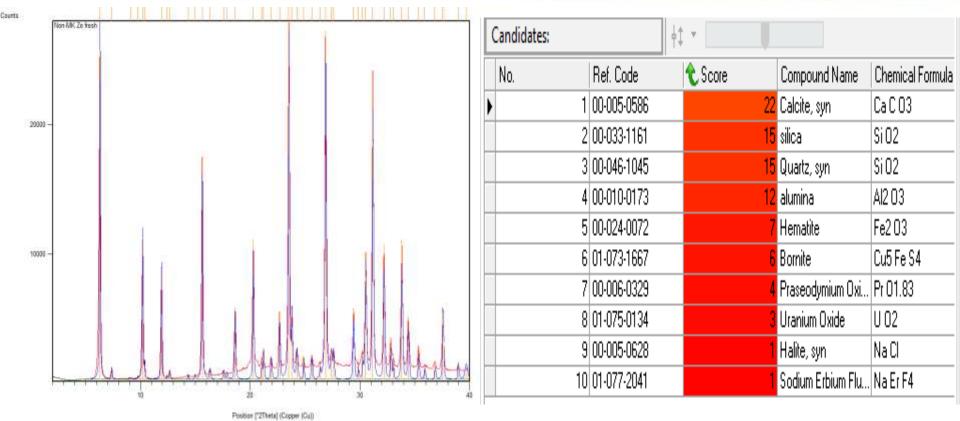
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- Presence of Calcite is considered significant
- Zeolite extrudate size ~4mm
- Si -Al ratio ~3 to 1
- Pore size unknown (TEM being considered)





LPA (TH-100) w/ 10% Cu Applied to Wire Mesh Trap

Un-coated wire mesh trap



Coated trap





DOC/DPF 143.8*152/300 5g/2:1:0

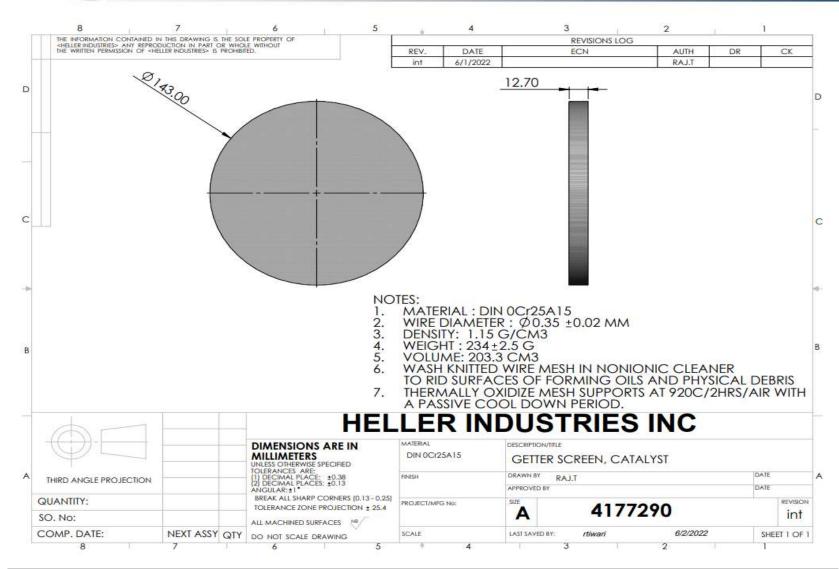
DOC/GPF 118.4*127/300 5g/2:1:0

CuSCR/GPF 118.4*127/300





Wire Mesh Trap Support





Atomic Size, Coking, Deactivation, Active Sites

Average zeolite is 1-10A Pore Diam.

10000A = 1 micron-U

C = 1.5A

O = 1.3A

H = 0.5A

H bond = 1.09A

- Silica/Alumina Ratio (SAR) will influence the acidity, thus activity
- Low SAR = more acidity with lower hydrophobicity, undesirable
- Higher Al results in greater coking and deactivation
- Too much acidity can lead to quicker coking and deactivation
- Many reactions require a tradeoff, (FAU and MFI)

Zeolyst: "some reactions can occur on the outer surface/pore mouth of a zeolite. so, molecules don't always need to fit in the pore".

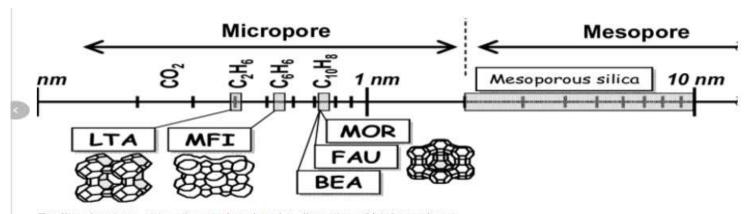


Zeolites – Pore Size

Pore Size	Number of Tetrahedra (MR 1)	Pore Diameter (Å)	Example
Small	8	4	PST-1 (NAT)
Medium	10	5.5	ZSM-5 (MFI)
Large	12	7.5	ZSM-12 (MTW)
Extra-large	>12	>7.5	CIT-5 (CFI)

¹ MR: Members of the ring.

Classification of zeolites according to their pore size.



Zeolite structure, pore size and molecular diameter of hydrocarbons.

Zeolyst: "some reactions can occur on the outer surface/pore mouth of a zeolite. so, molecules don't always need to fit in the pore"



Framework Type CHA

Framework 1

Cell Parameters: trigonal R -3 m (# 166)

a = 13.6750 Å b = 13.6750 Å c = 14.7670 Å $\alpha = 90.000^{\circ}$ $\beta = 90.000^{\circ}$ $y = 120.000^{\circ}$

Volume = 2391.6 A^3 $R_{DLS} = 0.0015$

Framework density (FD_{Si}): 15.1 T/1000 Å³

Topological density: \bigcirc TD₁₀ = 677 TD = 0.566667

Ring sizes (# T-atoms): 8 6 4

Channel dimensionality: 1 Topological (pore opening > 6-ring): 3-dimensional

Maximum diameter of a sphere: (1)

that can be included 7.37 Å

that can diffuse along a: 3.72 Å b: 3.72 Å c: 3.72 Å

Accessible volume: 17.27 %

ABC sequence AABBCC sequence of 6-rings

Secondary Building Units: 0 6-6 or 6 or 4-2 or 4

Composite Building Units: 0

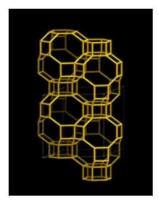


cha (t-cha)

d6r (t-hpr)

Natural Tiling (1) t-cha t-hpr

Framework images (click on icon for larger image)



Viewed normal to [001]



projection along [001]



Framework Type FAU

Framework

Cell Parameters: cubic F d -3 m (# 227)

 $a = 24.3450 \,\text{Å}$ $b = 24.3450 \,\text{Å}$ $c = 24.3450 \,\text{Å}$ $\alpha = 90.000^{\circ}$ $\beta = 90.000^{\circ}$ $y = 90.000^{\circ}$

Volume = 14428.8 Å^3 $R_{DLS} = 0.0009$

Framework density (FD_{Si}): 13.3 T/1000 Å³

Topological density: \bigcirc TD₁₀ = 579 TD = 0.476190

Ring sizes (# T-atoms): 12 6 4

Channel dimensionality: Topological (pore opening > 6-ring): 3-dimensional

Maximum diameter of a sphere: 0

that can be included 11.24 Å

that can diffuse along a: 7.35 Å b: 7.35 Å c: 7.35 Å

Accessible volume: 27.42 %

Secondary Building Units: 0 6-6 or 6-2 or 6 or 4-2 or 1-4-1 or 4

Composite Building Units: 0



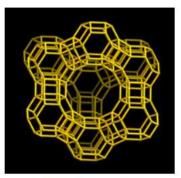
d6r (t-hpr)

Natural Tiling 10 t-fau t-hpr t-toc



sod (t-toc)

Framework images (click on icon for larger image)



Viewed along [111]



Polyhedral model viewed along [110]



an 'artist impression' of a catalytic reaction



viewed along [110]