



EMSL Analytical, Inc.

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Attn.: *Jim Cailahan*
WYK Sorbents, LLC
11721 Lackland Road
St.Louis, MO. 63146

EMSL Case No.: 361200001
Sample(s) Received: 12/30/11
Date of Analysis: 01/16/12
Date Printed: 01/16/12
Reported By: J.Newton
Email: jimc@wksorbents.com

Phone: 314-426-3336 Fax:


- Laboratory Report -

Project: Volcanic Ash Batch 1211A

Conclusions:

No evidence of fibrous erionite was detected during the analysis of samples 122711 (Volcanic Ash Batch 1211A).

Analyzed by:



John Newton
Senior Materials Scientist

16 January 2012

Date

Reviewed/Approved by :



Dana D'Ulisse
Approved Signatory



Eugenia Mirica, Ph.D.
Laboratory Manager

16 January 2012

Date



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Procurement of Samples and Analytical Overview:

The material arrived at EMSL Analytical's corporate laboratory in Cinnaminson, NJ on 12/30/11. The package arrived in satisfactory condition with no evidence of damage to the contents. The data reported herein has been obtained using the following equipment and methodologies.

Methods & Equipment: Scanning Electron Microscopy (SEM) – *JEOL, model JSM 6400*
Energy-dispersive X-Ray Spectrometry (EDX) – *EVAX, Nano*
X-Ray Diffraction Spectrometry (XRD) – *Rigaku, model DMAX 2200*

Definitions: Erionite: A naturally occurring fibrous variety of zeolite.
Fibrous: For the purposes of this study, particles with a minimum length to width ratio of 10:1.
Cleavage Fragment: For the purposes of this study, particles with a maximum length to width ratio of 10:1 and exhibiting bladed edges, tapered or non-parallel sides.

Background:

One sample consisting of volcanic ash was submitted for analysis. The focus of the analysis was to determine the concentration of the mineral Erionite in the specimen. The analysis was performed by scanning electron microscopy after preparation by gravimetric reduction to remove the organic component. The results of the study are documented in the following report.



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Summary of Results:

Sample ID	Description	Analyte	Concentration (%)	Comments
122711	Volcanic Ash Batch 1211A	Erionite	<LOQ	A,D
		Other Minerals:		
		Fibrous	<LOQ	B
		Cleavage Fragments	<LOQ	C
		Moisture & Organic Matter	5.2	E

Limit of Quantitation <0.1%

- Comments:
- A) Erionite minerals having the average chemical formula from Calcium-rich (Ca,K₂,Na₂)₂[Al₄Si₁₄O₃₆]•15(H₂O) to potassium-rich (K₂,Ca,Na₂)₂[Al₄Si₁₄O₃₆]•15(H₂O). Sodium was not present in sufficient quantity to assume sodium-rich erionite.
 - B) Particles exhibiting a fibrous habit but not elementally consistent with erionite.
 - C) Particles exhibiting bladed cross-section and/or tapered edges and therefore not a true fibrous morphology.
 - D) The Limit of Quantitation (LOQ) for the method is based upon particle counts and adjusted for the organic content.
 - E) Value obtained by gravimetric reduction (weight loss at 450°C).

Sample Preparation & Analysis:

The sample was homogenized by hand mixing. A sub-sample was analyzed for organic matter per ASTM D2974, test method C. The resulting non-combustible material was hand ground and sieved to <90µm. The resulting comminuted particulate was dispersed onto double-sided carbon tape and coated with 200Å to 300Å of gold to reduce the buildup of charged electrons during analysis.

Analysis was performed by scanning electron microscopy to determine the concentration of fibrous to non-fibrous particulate within the sample based upon a minimum 10:1 length to width ratio. Minerals exhibiting fibrous to pseudo-fibrous habit were analyzed by energy dispersive x-ray spectrometry to determine their elemental composition. Particles exhibiting a fibrous habit and applicable elemental composition are



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documented as minerals of interest (MOI's). Minerals not consistent with erionite are grouped according to morphology and include Fibrous, Cleavage Fragment and Non-Fibrous categories.

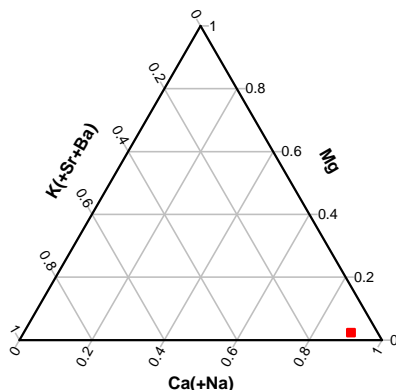
Minerals of interest matching the following criteria are documented as erionite.

- Fibrous Habit
- Magnesium cations (Mg^{2+}) less than or equal to 0.80 atoms per cell[†]
- Calculation of error (E%) $\pm 10\%$ [‡]
- Cation ratio $[Mg < 0.2] : [Ca(+Na) < 0.2] : [K(+Sr+Ba) < 0.8]$

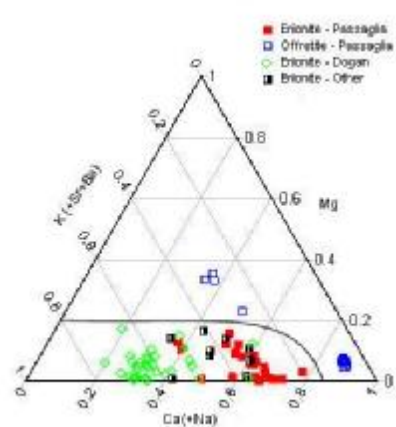
Results and Discussion:

The samples were composed predominantly of non-fibrous, lamellate to conchoidal grains. Some grains exhibited pseudo-fibrous morphology however, their elemental composition (alumino-silicate) is not consistent with erionite. Of the minerals exhibiting a pseudo-fibrous habit none maintained an elemental composition matching erionite[§]. Additional analysis by X-Ray Diffraction spectrometry (XRD) did not reveal any evidence of erionite or offretite.

Sample 122711 General Composition
Volcanic Ash Batch 1211A



Cation content for the pseudo-fibrous material observed in the sample



Combined compositional diagram showing cation content with proposed ranges for erionite vs. offretite.

[†] Passalia et al. 1998

[‡] Dogan et al. 2008

[§] Reference Doc.: Chemistry of Erionite and Offretite for Evaluation of Soil and Dust Samples in the Killdeer Region of North Dakota.

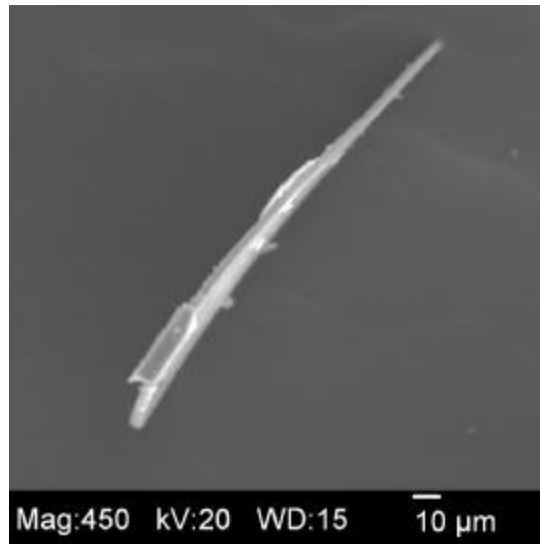


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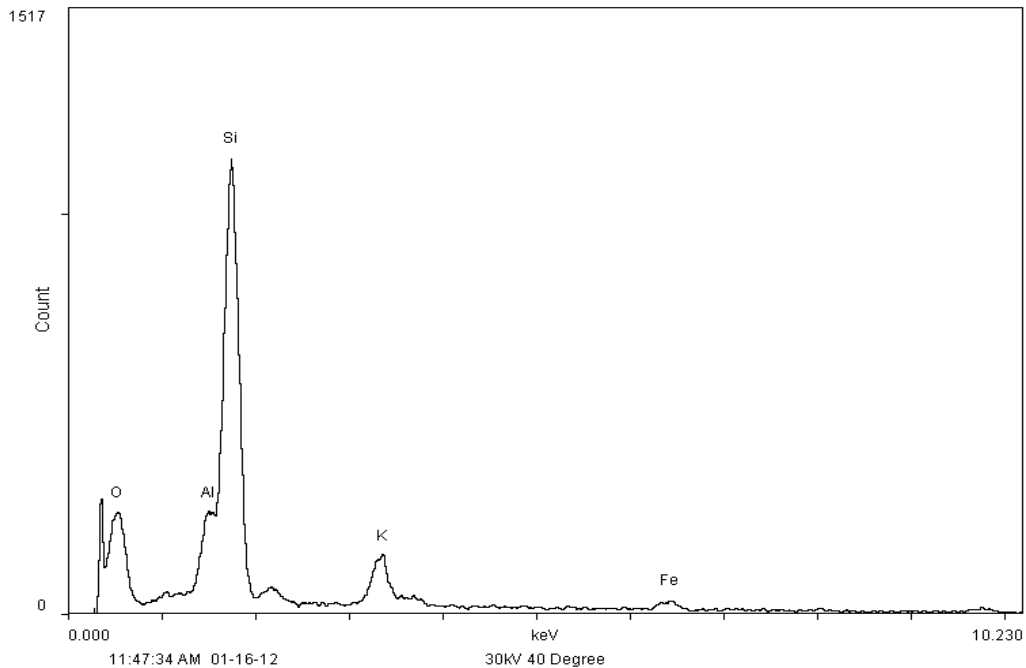
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Supporting Data:



SEM image of a pseudo-fibrous structure. This morphology is actually non-fibrous and is the result of the brittle ash fracturing and not a fibrous or acicular habit.



SEM/EDX spectrum showing the elemental composition of the structure.



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References

1. Dogan, A.U., Dogan, M., (2008) Re-evaluation and re-classification of erionite series minerals. Environ Geochem Health, 30:355-366
2. Passaglia, E., Artioli, G., Gualtieri, A. (1998). Crystal chemistry of the zeolites erionite and offretite, American Mineralogist 83, 577-589
3. Newton, J. (2009). Chemistry of Erionite and Offretite for Evaluation of Soil and Dust Samples in the Killdeer Region of North Dakota. Reference Document

Important Terms, Conditions, and Limitations:

Sample Retention: Samples analyzed by EMSL will be retained for 60 days after analysis date. Storage beyond this period is available for a fee with written request prior to the initial 30 day period. Samples containing hazardous/toxic substances which require special handling may be returned to the client immediately. EMSL reserves the right to charge a sample disposal or return shipping fee.

Change Orders and Cancellation: All changes in the scope of work or turnaround time requested by the client after sample acceptance must be made in writing and confirmed in writing by EMSL. If requested changes result in a change in cost the client must accept payment responsibility. In the event work is cancelled by a client, EMSL will complete work in progress and invoice for work completed to the point of cancellation notice. EMSL is not responsible for holding times that are exceeded due to such changes.

Warranty: EMSL warrants to its clients that all services provided hereunder shall be performed in accordance with established and recognized analytical testing procedures and with reasonable care in accordance with applicable federal, state and local laws. The foregoing express warranty is exclusive and is given in lieu of all other warranties, expressed or implied. EMSL disclaims any other warranties, express or implied, including a warranty of fitness for particular purpose and warranty of merchantability.

Limits of Liability: In no event shall EMSL be liable for indirect, special, consequential, or incidental damages, including, but not limited to, damages for loss of profit or goodwill regardless of the negligence (either sole or concurrent) of EMSL and whether EMSL has been informed of the possibility of such damages, arising out of or in connection with EMSL's services thereunder or the delivery, use, reliance upon or interpretation of test results by client or any third party. We accept no legal responsibility for the purposes for which the client uses the test results. EMSL will not be held responsible for the improper selection of sampling devices even if we supply the device to the user. The user of the sampling device has the sole responsibility to select the proper sampler and sampling conditions to insure that a valid sample is taken for analysis. Any resampling performed will be at the sole discretion of EMSL, the cost of which shall be limited to the reasonable value of the original sample delivery group (SDG) samples. In no event shall EMSL be liable to a client or any third party, whether based upon theories of tort, contract or any other legal or equitable theory, in excess of the amount paid to EMSL by client thereunder.

The data and other information contained in this report, as well as any accompanying documents, represent only the samples analyzed. They are reported upon the condition that they are not to be reproduced wholly or in part for advertising or other purposes without the written approval from the laboratory.