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TO

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SUBJ XRF analysis of Lumber Samples to Detect Copper from Preservative Treatments

Summary

Disintegrating Douglas fir lumber from a bridge and pieces of untreated cedar wood and of known preservative pressure treated wood in the AME facility were analyzed using XRF (X-Ray Fluorescence) to determine their elemental composition for carbon, nitrogen, fluorine, sodium, magnesium, aluminum, silicon, and all heavier elements. We were tasked with determining whether the wood had been treated with a copper-based wood preservative. There was particular interest in copper since it is found in the common non-organic wood preservatives. Some of the organic preservatives such as creosote can be visually ruled out.

- Two different pieces of lumber from the bridge had ~0.29 wt% Cu and ~0.69 wt% of copper (Cu), respectively.
- The cedar wood, which was thought to be untreated with preservative, had no detectable copper in it.
- The known pressure treated wood had ~0.36 wt% of copper in it.
- Thus, the lumber from the bridge seems to be pressure treated wood.
- Both of the disintegrating bridge wood samples had silicon (Si) concentrations that were very high. These concentrations were ten times higher than the two AME wood samples used as reference wood samples.

Samples and Background

Wood samples identified as from the surfaces of rotting wood from a bridge were sent for analysis for a wood preservative. See Figure 1 for a picture of the wood samples we received for analysis. Figure 2 shows the samples which were actually analyzed by XRF with the surface facing the x-ray source and detector shown.

Figure 1. The disintegrating wood received from the surfaces of the bridge wood.

Figure 2. The two samples taken from the submitted bridge wood sample material which were analyzed by XRF are shown at the top of this picture. The cedar wood sample is at the lower left and the known pressure treated preservative wood sample with the slightly green surface color is shown in the lower right of the picture.

XRF Spectrometry Analysis

Our wavelength-dispersive XRF spectrometer can quantitatively measures the elemental concentrations for all elements from fluorine through uranium and when the material has a low density, as in polymers, we can also analyze carbon and nitrogen using an additional crystal. The depth of analysis depends upon the characteristic x-ray energy emitted from the detected element and the density of the material. This depth can vary from a micrometer to a millimeter. XRF analysis has very low detection limits for the elements. Wavelength-dispersive XRF systems have greater elemental sensitivity and higher energy resolution than do less expensive energy-dispersive XRF spectrometers. We can detect all but the lightest elements at concentrations as low as 10 ppm. Solid Samples, powders, and liquids can be analyzed with XRF analysis. Our spectrometer also has an unusual small spot capability to measure spots of 0.5 or 1.5-mm diameter, as well as the capability to measure areas of 10 mm and 29 mm diameter. Of course, large area measurements offer lower detection limits and greater accuracy of measurement. For this work, the 29 mm aperture was used, and the Samples were analyzed in vacuum.

Figures 3-6 show the elemental composition analysis of lumber pieces from bridge, cedar wood and a pressure treated wood. The two different pieces of lumber from the bridge had ~0.29 wt% copper (Cu) and ~0.69 wt% Cu, respectively. The cedar wood had no Cu in it while the pressure treated wood had ~0.36 wt% Cu in it. Thus, the lumber from the bridge has a copper concentration consistent with that of wood treated with a copper-based wood preservative.

There is a curious observation to be made. Both of the disintegrating bridge wood samples had silicon (Si) concentrations that were very high. These concentrations were ten times higher than the two AME wood samples used as reference wood samples. What is the cause of such a high Si concentration?

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1/12/2024 3:35:59PM
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Thermo Fisher Scientific

Douglas FIR Lumber - SHL

PFX-099 Rh 60kV LiF200 LiF220 Ge111 AX03
Method : X_UQi with CN
Kappa List : AnySample
Shapes & ImpFc : Teflon
Calculated as : Elements
Case Number : 0 = All known

Measure time : 1/9/2024 12:17:06PM
X-ray Path: : Vacuum
Film Type : None
Collimator Mask : 29 mm
Viewed Diameter = 29.00 mm
Viewed Area = 660.52 mm2
Viewed Mass = 1576.88 mg
Sample Height = 2.46 mm

Reporting Level > 10 ppm and wt% > 3 Est.Err.

Element	Wt%	Est.Error
C	79.46	0.20
Si	6.78	0.13
N	5.95	0.12
Al	3.28	0.09
Fe	1.52	0.06
K	0.951	0.047
Mg	0.356	0.018
Na	0.318	0.035
Cu	0.293	0.015
Ti	0.268	0.013
Ca	0.256	0.023
S	0.253	0.013
Px	0.153	0.0077
Ba	0.0401	0.0097
Cl	0.0330	0.0017
Cs	0.0230	0.0074
Mn	0.0200	0.0010
I	0.0173	0.0044
Zn	0.0121	0.0006
Zr	0.0077	0.0010
Cr	0.0054	0.0004
V	0.0043	0.0004
Sr	0.0031	0.0008

Sum Weight% before normalization to 100% = 48.7 %

Figure 3. Elemental composition analysis of Lumber from bridge decline – piece 1 using WD XRF. Note the high silicon (Si) concentration.

Anderson Materials Eva. - US

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Thermo Fisher Scientific

Bridge wood_2 -

PFX-099 Rh 60kV LiF200 LiF220 Ge111 AX03
Method : X_UQi with CN
Kappa List : AnySample
Shapes & ImpFc : Teflon
Calculated as : Elements
Case Number : 0 = All known

Measure time : 1/11/2024 2:24:38PM
X-ray Path: : Vacuum
Film Type : None
Collimator Mask : 29 mm
Viewed Diameter = 29.00 mm
Viewed Area = 660.52 mm2
Viewed Mass = 1576.88 mg
Sample Height = 2.46 mm

Reporting Level > 10 ppm and wt% > 3 Est.Err.

Element	Wt%	Est.Error
C	80.37	0.20
Si	6.24	0.12
N	6.12	0.12
Al	3.03	0.09
Fe	1.18	0.05
K	0.711	0.035
Cu	0.685	0.034
Na	0.346	0.038
Mg	0.336	0.017
Ti	0.228	0.011
Ca	0.223	0.020
S	0.196	0.0098
Px	0.127	0.0063
Cl	0.0514	0.0026
Ba	0.0485	0.013
Cs	0.0328	0.0099
I	0.0210	0.0059
Cr	0.0180	0.0009
Mn	0.0163	0.0008
Zr	0.0076	0.0012
V	0.0032	0.0004
Zn	0.0026	0.0005

Sum Weight% before normalization to 100% = 48.6 %

Figure 4. Elemental composition analysis of Lumber from bridge decline – piece 2 using WD XRF. Note the high Si concentration.

Anderson Materials Eva. - US

1/12/2024 3:39:01PM
Calculated by UniQuant
Thermo Fisher Scientific

Cedar Wood - AME

PFX-099 Rh 60kV LiF200 LiF220 Ge111 AX03
Method : X_UQi with CN
Kappa List : AnySample
Shapes & ImpFc : Teflon
Calculated as : Elements
Case Number : 0 = All known

Measure time : 1/9/2024 7:30:33PM
X-ray Path: : Vacuum
Film Type : None
Collimator Mask : 29 mm
Viewed Diameter = 29.00 mm
Viewed Area = 660.52 mm2
Viewed Mass = 1576.88 mg
Sample Height = 2.50 mm

Reporting Level > 10 ppm and wt% > 3 Est.Err.

Element	Wt%	Est.Error
C	91.67	0.14
N	6.89	0.13
Si	0.600	0.030
Al	0.201	0.010
Ca	0.170	0.015
F	0.110	0.035
Sx	0.0783	0.0039
Na	0.0661	0.0073
Mg	0.0539	0.0027
Fe	0.0406	0.0020
Ba	0.0278	0.0078
K	0.0216	0.0011
Cs	0.0185	0.0060
Cl	0.0168	0.0008
Zn	0.0141	0.0007
Ti	0.0072	0.0004
Px	0.0061	0.0003
Cr	0.0056	0.0003

Sum Weight% before normalization to 100% = 46.9 %

Figure 5. Elemental composition analysis of cedar wood using WD XRF. Note that no copper (Cu) was detected and the Si concentration is only about one-tenth that of the rotting bridge wood.

Anderson Materials Eva. - US

1/12/2024 3:40:53PM
Calculated by UniQuant
Thermo Fisher Scientific

Pressure treated wood - AME

PFX-099 Rh 60kV LiF200 LiF220 Ge111 AX03
Method : X_UQi with CN
Kappa List : AnySample
Shapes & ImpFc : Teflon
Calculated as : Elements
Case Number : 0 = All known

Measure time : 1/11/2024 7:57:30PM
X-ray Path: : Vacuum
Film Type : None
Collimator Mask : 29 mm
Viewed Diameter = 29.00 mm
Viewed Area = 660.52 mm2
Viewed Mass = 11563.75 mg
Sample Height = 4.00 mm

Reporting Level > 10 ppm and wt% > 3 Est.Err.

Element	Wt%	Est.Error
C	84.29	0.18
N	10.64	0.15
Cr	1.45	0.06
Si	0.554	0.028
As	0.518	0.026
Ca	0.457	0.041
Al	0.414	0.021
Cu	0.364	0.018
Sx	0.286	0.014
Px	0.263	0.013
Na	0.250	0.028
Mg	0.146	0.0073
K	0.138	0.0069
Cl	0.102	0.0051
Fe	0.0649	0.0032
Zn	0.0286	0.0014
Mn	0.0214	0.0011
Ti	0.0116	0.0006
Ce	0.0075	0.0020

Sum Weight% before normalization to 100% = 44.3 %

Figure 6. Elemental composition analysis of a pressure treated wood using WD XRF. Note that the Cu concentration is 0.36 wt.% and that the Si concentration is less than one-tenth that of the rotting bridge wood.