Academy of Scientific Research And Technology
أكاديمية البحث العلمي والتكنولوجيا

FLNP

Raman micro spectroscopy

Presented By:

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15.05-02.06.2023

Supervisor

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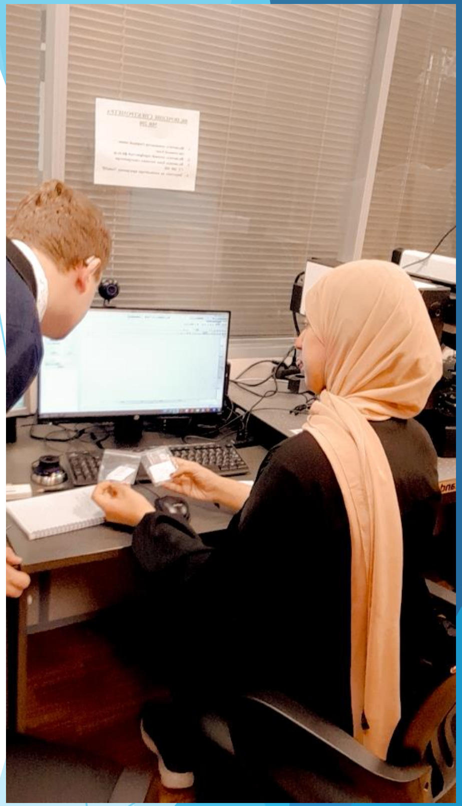
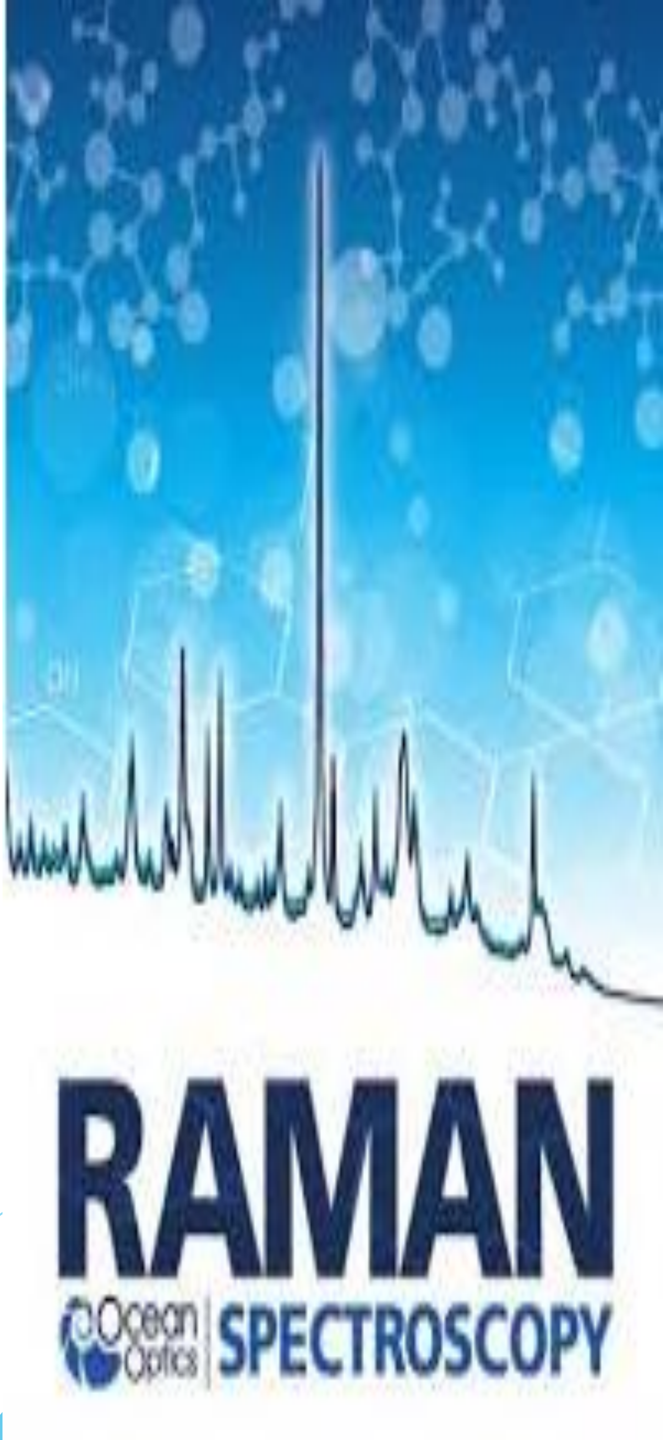


Outlines

- *Introduction*
- *Aims of the work*
- *Principle of Raman spectroscopy*
- *How to make measurements?*
- *Raman spectrum*
- *Conclusion*



- Spectroscopic methods of analysis always afford reliable methods for identification of the substances.
- A condition for the material to be infrared active, a change in dipole moment If there is no change in the dipole moment, the material becomes infrared inactive so I need anther technique to know molecular structure, so we use the Raman technique.

- The natural microsphere using to remove the heavy metal from the wastewater
- The fig indicated the model structure for Sodium alginate/water hyacinth composite in the form of microsphere

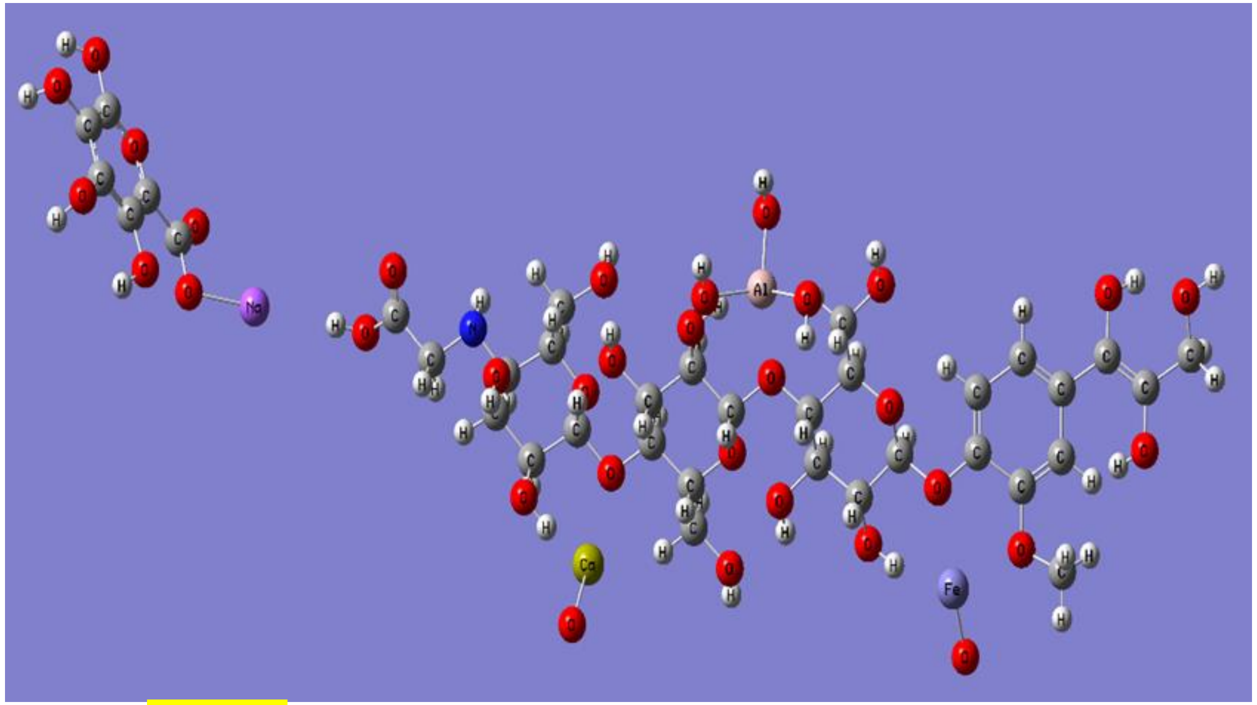


figure :indicated the model structure of the microsphere

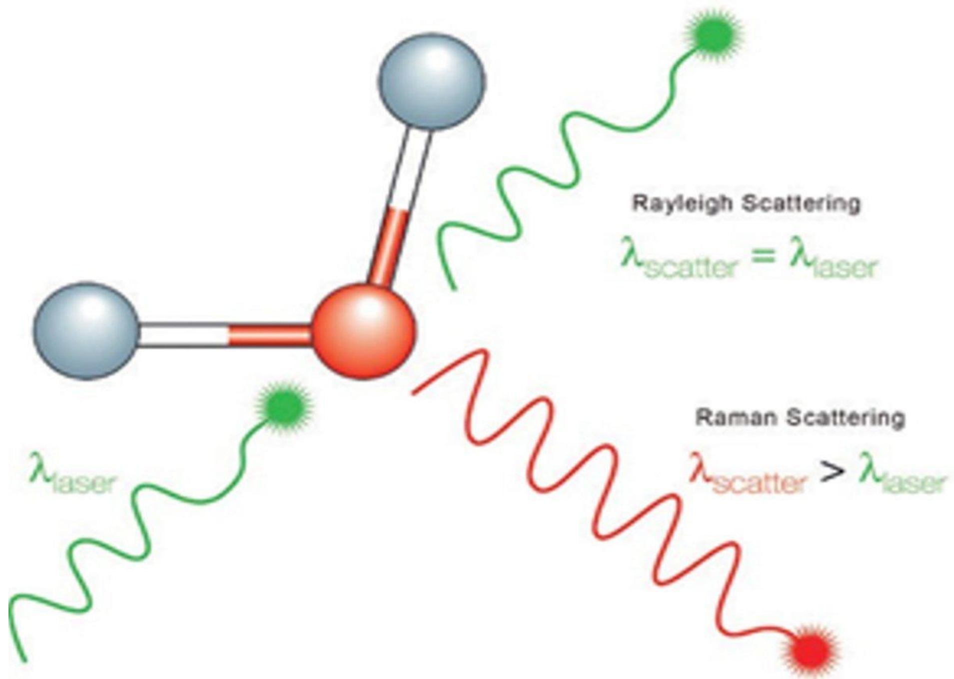
Aims of the work

- Introduction to Raman spectroscopy and learning to work on.
- Application of Raman spectroscopy to determine the presence of lead oxides on the surface of the microsphere, depending on the holding time in solution.
- Processing of the obtained Raman spectra in the OriginPro-2021 program.
- Determine the structure of lead oxide from the Raman peak. (Analysis of Raman spectra).

Principles of Raman spectroscopy

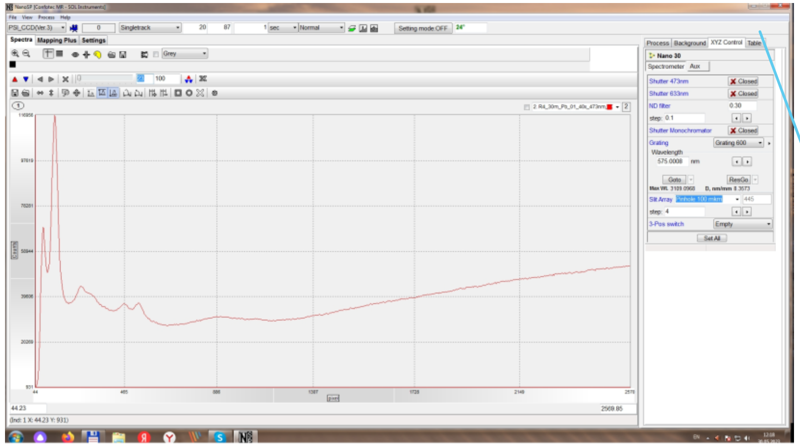
Raman Spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure, phase and crystallinity and molecular interactions.

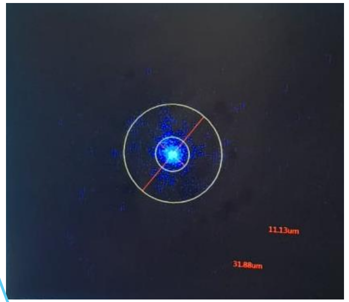
It is based upon the interaction of light with the chemical bonds within a material.

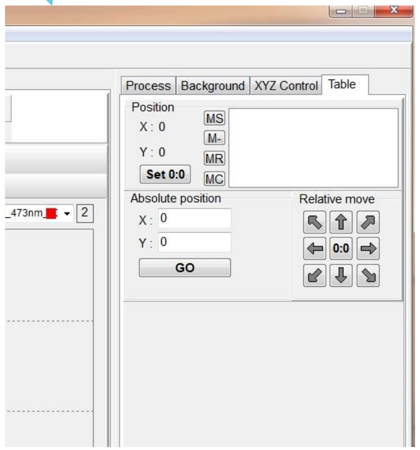


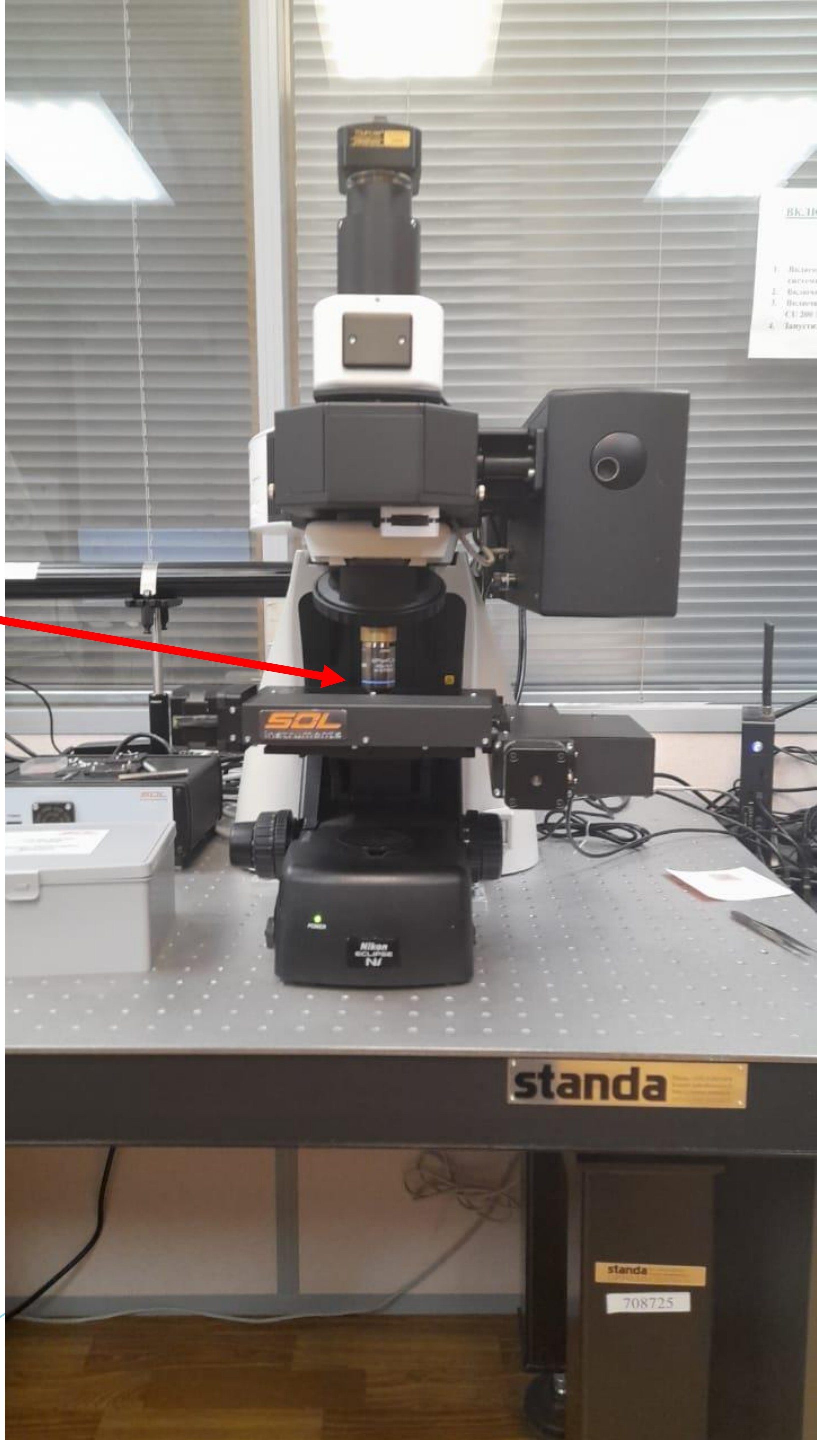
How to make measurements?

Sample



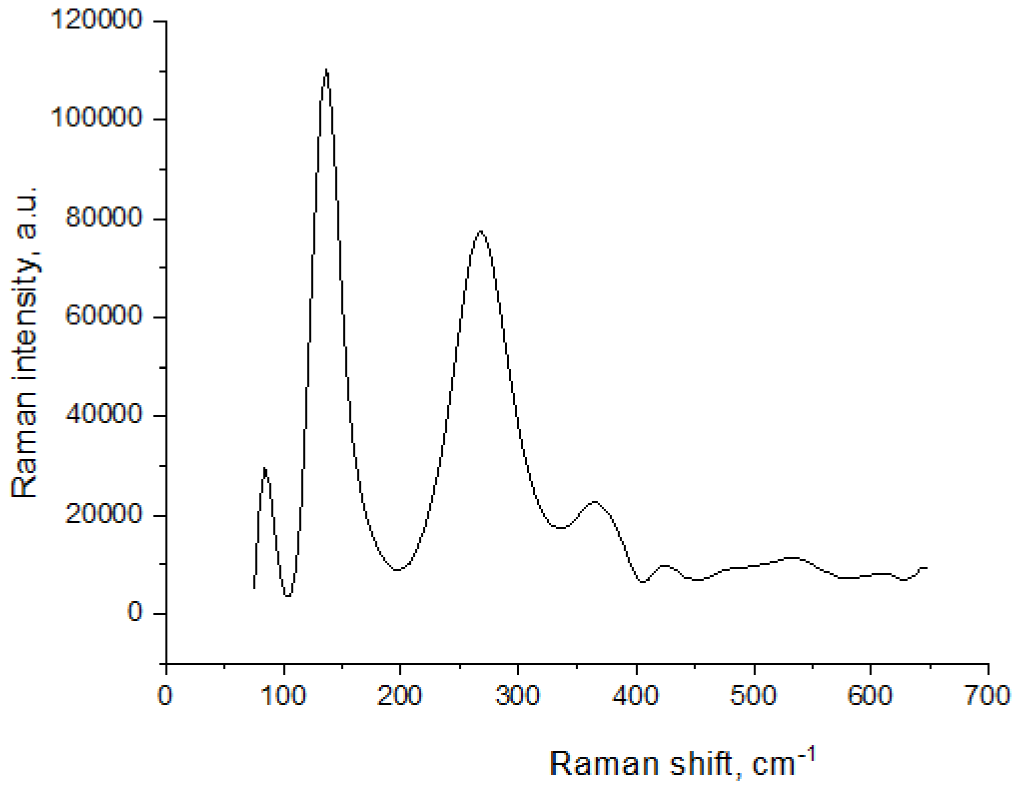


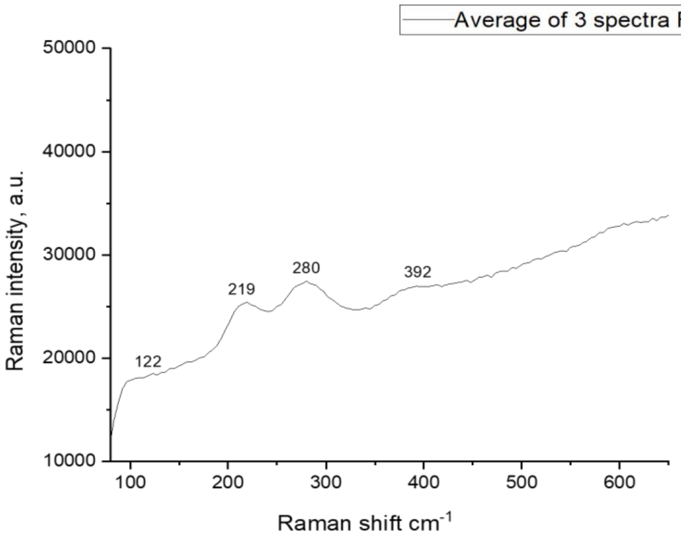




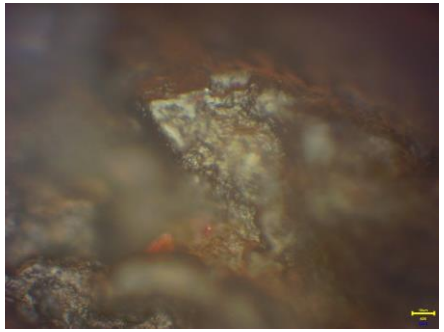
Raman spectrum

A Raman spectrum is a plot of the intensity of Raman scattered radiation as function of its frequency difference from the incident radiation . This difference is called the Raman shift.

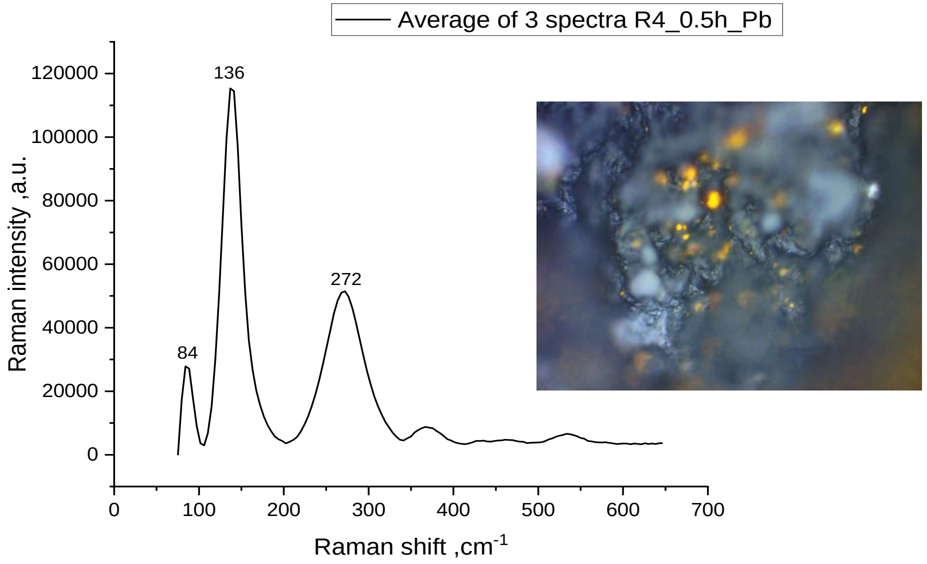



Average of 3 spectra R4 0h
Raman intensity, a.u.
Raman shift cm^{-1}
122
219
280
392
10000
20000
30000
40000
50000
100
200
300
400
500
600

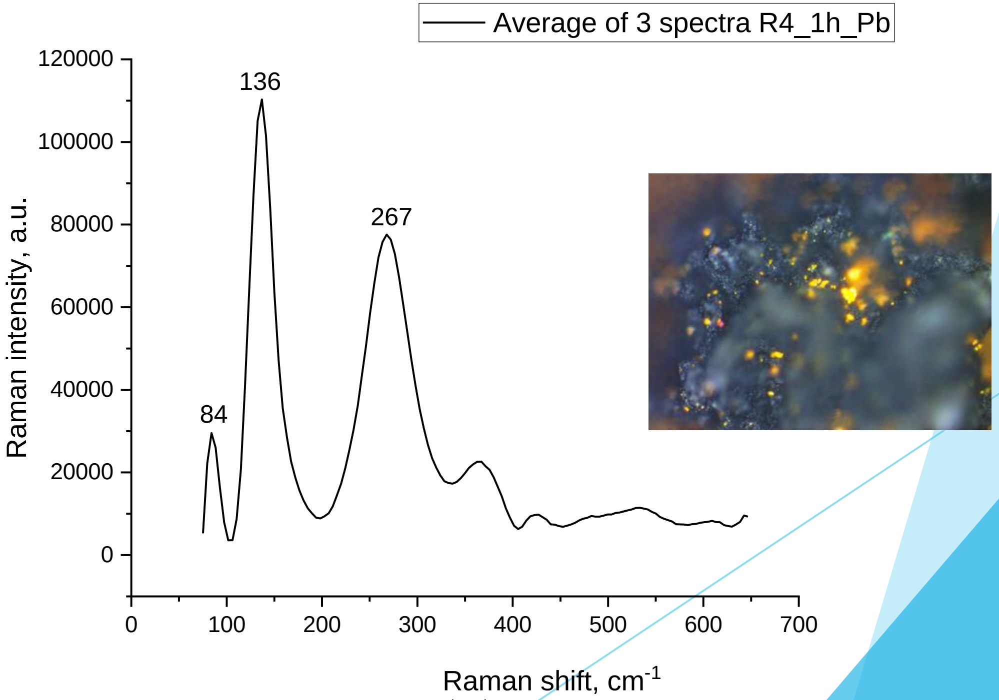
R4_Pb_0h



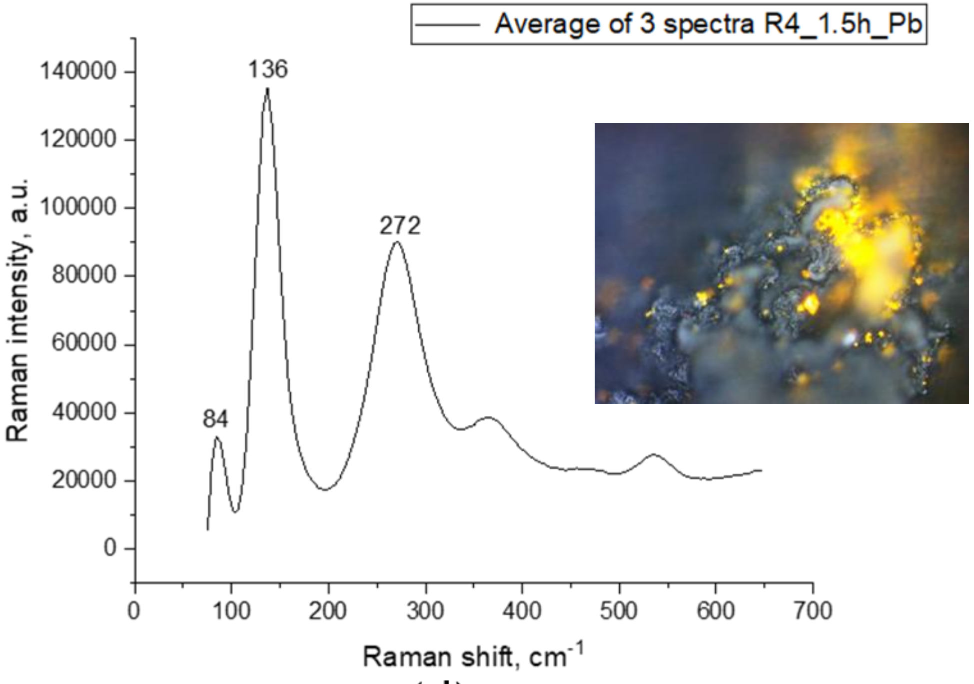
(a)


Average of 3 spectra R4_0.5h_Pb
Raman intensity ,a.u.
Raman shift ,cm^{-1}
84
136
272
0
20000
40000
60000
80000
100000
120000
0
100
200
300
400
500
600
700

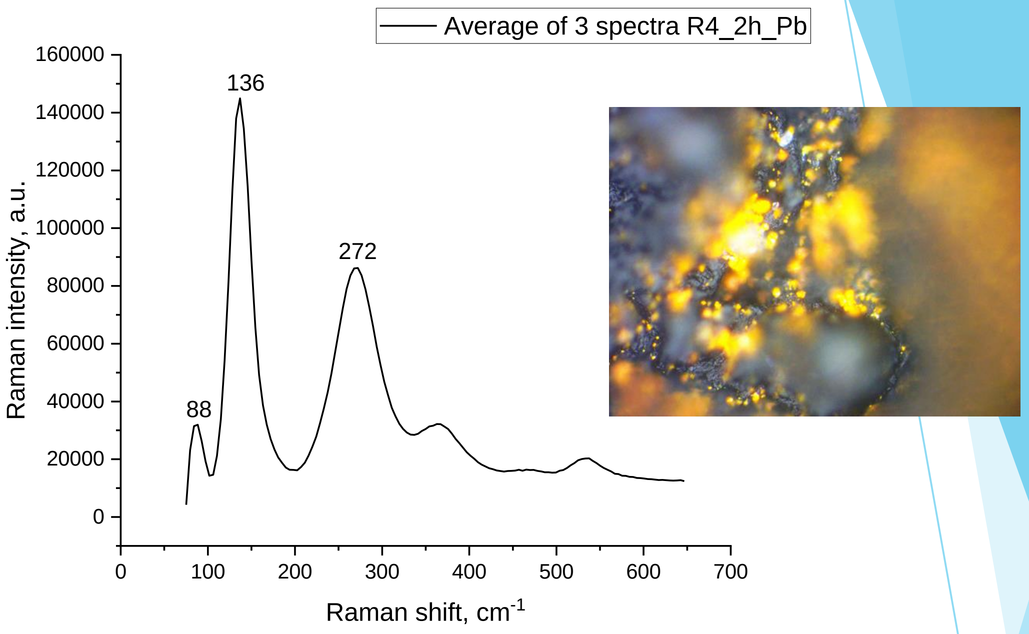
(b)


Average of 3 spectra R4_1h_Pb
Raman intensity, a.u.
Raman shift, cm^{-1}
84
136
267
0
20000
40000
60000
80000
100000
120000
0
100
200
300
400
500
600
700

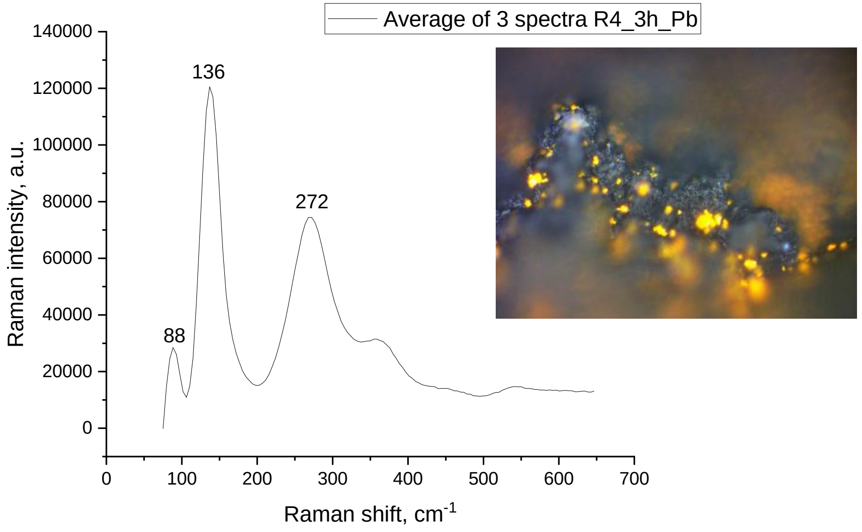
(c)


Average of 3 spectra R4_1.5h_Pb
Raman intensity, a.u.
Raman shift, cm^{-1}
84
136
272
0
20000
40000
60000
80000
100000
120000
140000
0
100
200
300
400
500
600
700

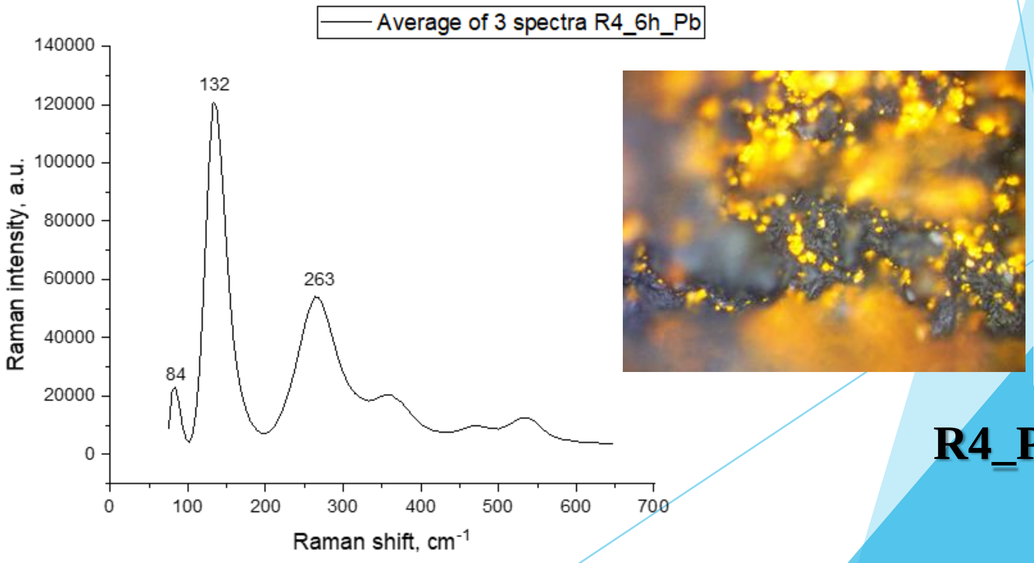
(d)


Average of 3 spectra R4_2h_Pb
Raman intensity, a.u.
Raman shift, cm^{-1}
88
136
272
0
20000
40000
60000
80000
100000
120000
140000
160000
0
100
200
300
400
500
600
700

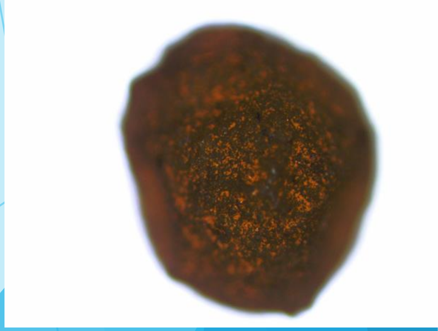
(e)


Average of 3 spectra R4_3h_Pb
Raman intensity, a.u.
Raman shift, cm^{-1}
88
136
272
0
20000
40000
60000
80000
100000
120000
140000
0
100
200
300
400
500
600
700

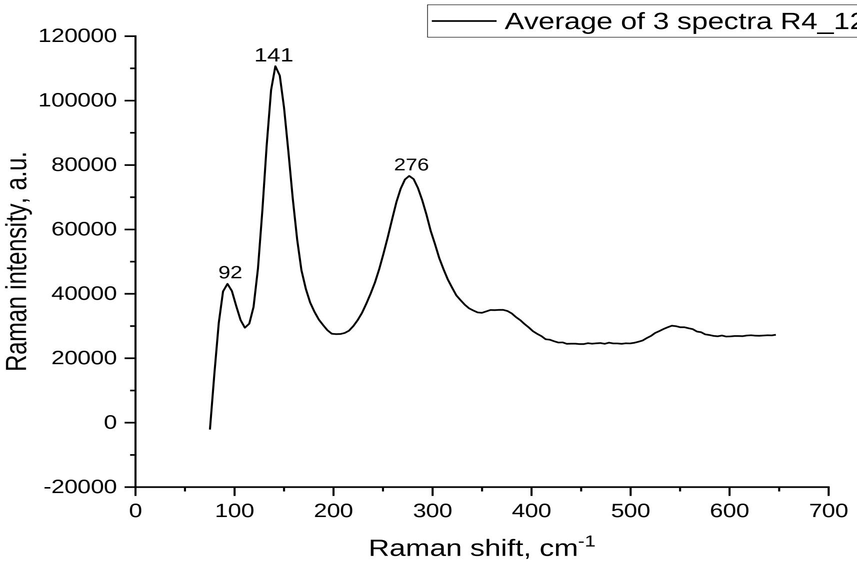
(f)


Average of 3 spectra R4_6h_Pb
Raman intensity, a.u.
Raman shift, cm^{-1}
84
132
263
0
20000
40000
60000
80000
100000
120000
140000
0
100
200
300
400
500
600
700

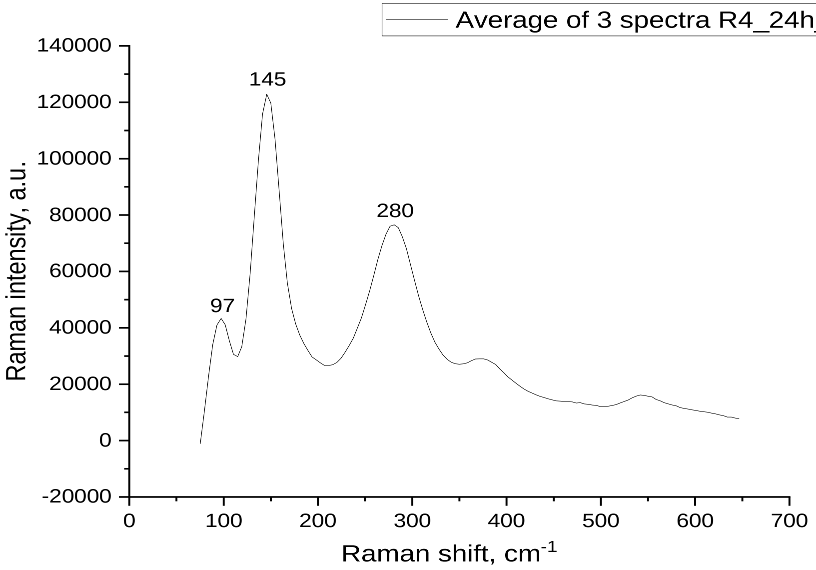
(g)



R4_Pb_6h

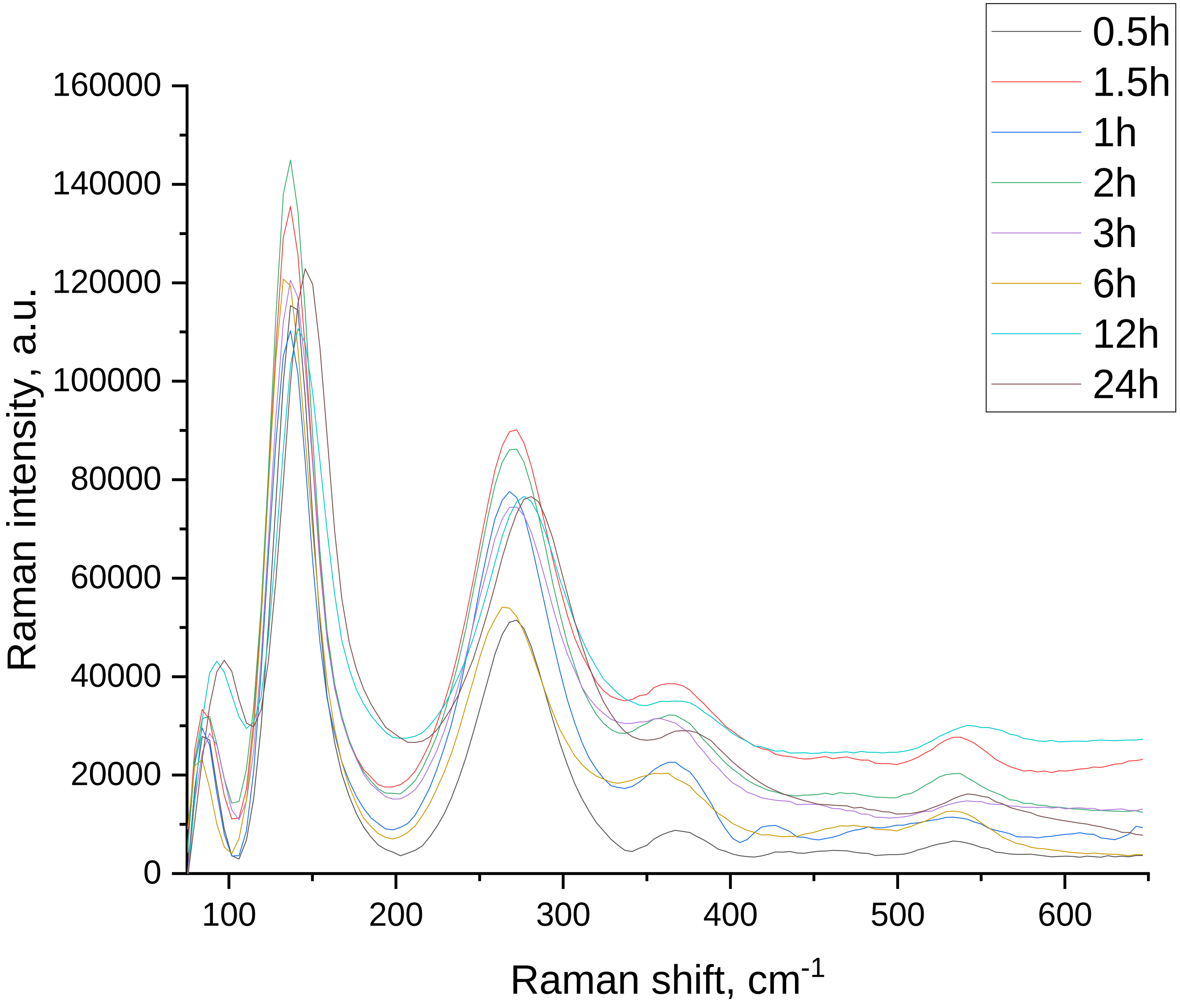

Average of 3 spectra R4_12h_Pb
Raman intensity, a.u.
Raman shift, cm-1
141
276
92
120000
100000
80000
60000
40000
20000
0
-20000
0
100
200
300
400
500
600
700

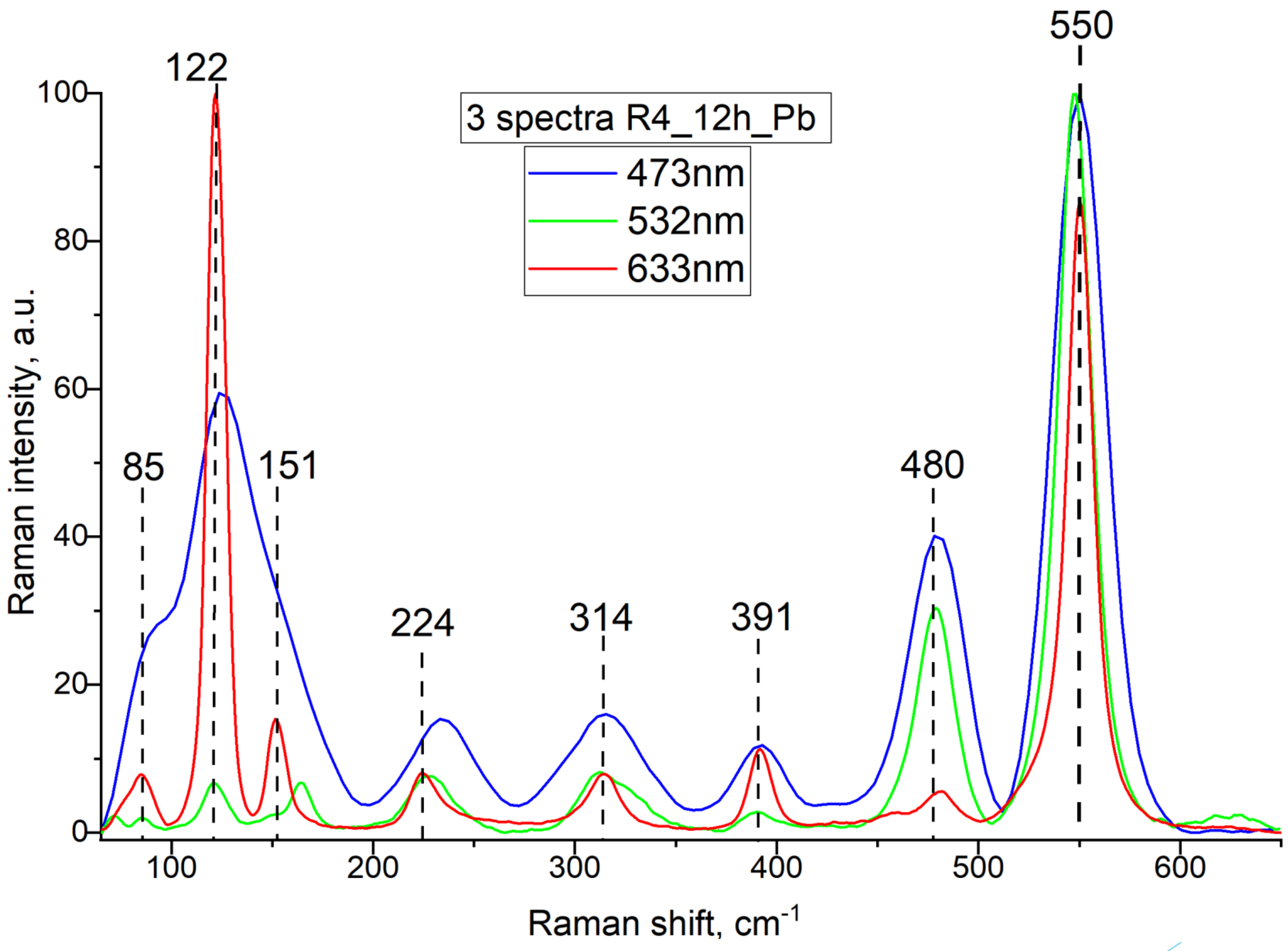
(h)


Average of 3 spectra R4_24h_Pb
Raman intensity, a.u.
Raman shift, cm-1
145
280
97
140000
120000
100000
80000
60000
40000
20000
0
-20000
0
100
200
300
400
500
600
700

R4_Pb_24h

(i)


0.5h
1.5h
1h
2h
3h
6h
12h
24h
160000
140000
120000
100000
80000
60000
40000
20000
0
100
200
300
400
500
600
Raman intensity, a.u.
Raman shift, cm-1



3 spectra R4_12h_Pb
473nm
532nm
633nm
122
550
85
151
224
314
391
480
Raman intensity, a.u.
Raman shift, cm-1
0
20
40
60
80
100
100
200
300
400
500
600

- we also measure at 3 different wavelength. Results shows that we obtained all main peaks of lead oxide at each wavelength of laser excitation. Best results give us at 532nm and 633nm excitation, because we get more detailed peaks in low frequency region (88-200 cm^{-1}).

this all 3 spectra were taken under lower laser power of excitation, for do not damage the samples.

table of assignments from literature for info:

Table 1. Raman bands (cm^{-1}) and correspondence found on the wall paintings samples while varying the laser power with the density of absorbing neutral filters D2, D0.6, D0.0 and D2.

Compounds	D2	D0.6	D0.0	D2
Plattnerite (β- PbO_2)	515			
Scrutiniyte (α-PbO_2)	82, 165, 228			
Red lead Pb_3O_4			116 (shi), 534 (shi)	122, 548
Litharge (α-PbO)		77 (shi)	77 (shi), 328 (shi)	80, 146, 336
Massicot (β-PbO)		138 (br/shi)	137 (br/shi), 81 (shi), 270 (shi)	85, 142 (sh), 285

shoulder (sh), broad (br), shifted (shi).

Conclusion

- We obtained that Raman spectroscopy could be applied for detecting lead oxide on the microspheres depend on time. Microphotography also confirms that with increasing time the amount of lead oxides on the samples also increases, and this is confirmed by Raman spectroscopy.
- According to the analysis of the obtained Raman spectrum, it was found that mainly in the mixture is formed lead oxides such as:
  - Scrutiniyte (PbO_2) — at 82, 165, 228cm^{-1}
  - Red lead(Pb_3O_4)— at 122, 548cm^{-1}(main contribution — more intensive picks)
  - Litharge (PbO) — at 80, 146cm^{-1}
  - Massicot (PbO) — 85, 137-142, 270-280cm^{-1}(if damaged by laser)

Thank You