Global Journal of Engineering and Technology Advances
eISSN: 2582-5003
Cross Ref DOI: 10.30574/gjeta
Journal homepage: https://gjeta.com/

(RESEARCH ARTICLE)

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Dispersion and Morphological studies of Single-walled carbon nanotubes using Sodium Dodecyl Sulphate as a Surfactant

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Global Journal of Engineering and Technology Advances, 2025, 23(03), 242-263

Publication history: Received on 09 May 2025; revised on 15 June 2025; accepted on 17 June 2025

Article DOI: https://doi.org/10.30574/gjeta.2025.23.3.0202

Abstract

The sonication-driven dispersion of single-walled carbon nanotubes (SWCNTs) in aqueous surfactant (SDS) solution has been monitored by UV-visible spectroscopy and scanning electron microscopy (SEM). Dispersion of SWCNTs experiments revealed that the Sonication time and dilution factor of dispersed SWCNTs affect the UV-visible absorbance of the solution. With optimal surfactant concentration, the dispersion rate of SWCNTs increased, and low-temperature (24°C) sonication was required to achieve maximum dispersion. Dispersion of higher SWCNT concentrations requires longer sonication time. Morphological surface study of single-walled carbon nanotubes (SWCNTs) solution gives the result of surface orientation analyzed by DEKTAK surface profiler, whether SWCNTs are uniformly orientated on the surface and also homogeneously dispersed into the solution or not.

Keywords: Single-Walled Carbon Nanotubes (SWCNTs); Sodium Dodecyl Sulphate (SDS); UV-Visible Absorption Spectra; Dektak Vecco 150 Surface Profiler; Scanning Electron Microscopy (SEM)

1. Introduction

In the last decade, researchers have been interested in the unique biochemical and mechanical properties of single-walled carbon nanotubes (SWCNTs)[1-3]However, their smooth, highly polarized surfaces and van der Waals forces tend to fold, making them insoluble in conventional solvents and water. Their usefulness was limited for that reason [4, 5]. Consequently, obtaining efficient dispersions of SWCNTs has become an important task.

Several methods have been explored to enhance the dispersion of SWCNT, mainly by surface modification using different surfactants, including anionic, cationic, and non-ionic species such as sodium deoxycholate (DOC)[6], sodium dodecylbenzene sulfate (SDBS)[7], sodium dodecyl sulfate (SDS)[8] and the family of tween surfactant[9]. Techniques such as atomic force microscopy (AFM)[1, 10], Raman spectroscopy[11-13], Scanning Electron Microscopy (SEM)[14, 15], cryogenic transmission electron microscopy (cryo-TEM)[16], Dektak Vecco 150 Surface profiler can be used to study SWCNT dispersions[17-19].

However, a standardized method for monitoring its prevalence remains elusive. The need for a simple but reliable method is important in practical design. UV-Visible spectroscopy has emerged as a promising tool to evaluate SWCNT dispersion[20]. In the UV-Visible region, individual SWCNTs exhibit distinct absorption bands attributed to 1D van Hove singularities[21]. Bundled SWCNTs exhibit little activity at this wavelength due to carrier tunneling between

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nanotubes[22]. The researchers noted that the maximum absorbance is between 200 and 300 nm, which gradually decreases from the UV to the near IR. Consequently, it appears that the concentration of individually dispersed SWCNTs in a solution can be correlated with the intensity of the corresponding absorption spectrum. This paper proposes a simplistic and convenient way by using UV-vis-NIR absorbance, Dektak Vecco 150 Surface profiler, and Scanning Electron Microscopy (SEM) to assess dispersion efficacy and leveraging it as a benchmark for identifying optimal conditions along with uniform distribution of SWCNT dispersion in surfactant solutions which could be used in a solar cell matrix, a sensor such as gas sensor[23-30], etc. and also this systematic study leaves much scope for future investigation Such as Study of dispersion of multi-walled carbon nanotubes(MWCNTs) and also use of different method with different surfactant solution.

2. Materials and Methods

2.1. Solution Preparation and Dispersion

Single-walled carbon nanotubes (SWCNTs) were purchased from (Hipco, Sigma-Aldrich, 0.7 - 1.1 nm). The surfactant for the dispersion of the SWCNTs was sodium dodecyl sulfate (SDS; 99%) purchased from Sigma-Aldrich. All dispersion experiments were carried out with distilled water. There were several steps in the dispersion process. Firstly, the SDS solution concentration was used as 1.5 wt%[31, 32] and it was prepared by 0.299g of SDS and dissolved in 19.7 ml of DI water. Secondly, the SWCNTs Solution was prepared by mixing the SDS (1.5 wt%) solution with SWCNTs in a matric ratio, and the ratio we used 5 ml SDS solution for 0.3mg SWCNTs. Finally, The Dispersion of SWCNTs in SDS solution was performed in a test tube, and the test tube was placed in a digital ultrasonic bath (UBT 580) for 2 Hours of sonication with a controlled temperature of 24°C by using an Ice Cube[32, 33].

2.2. Sample Preparation for Characterization

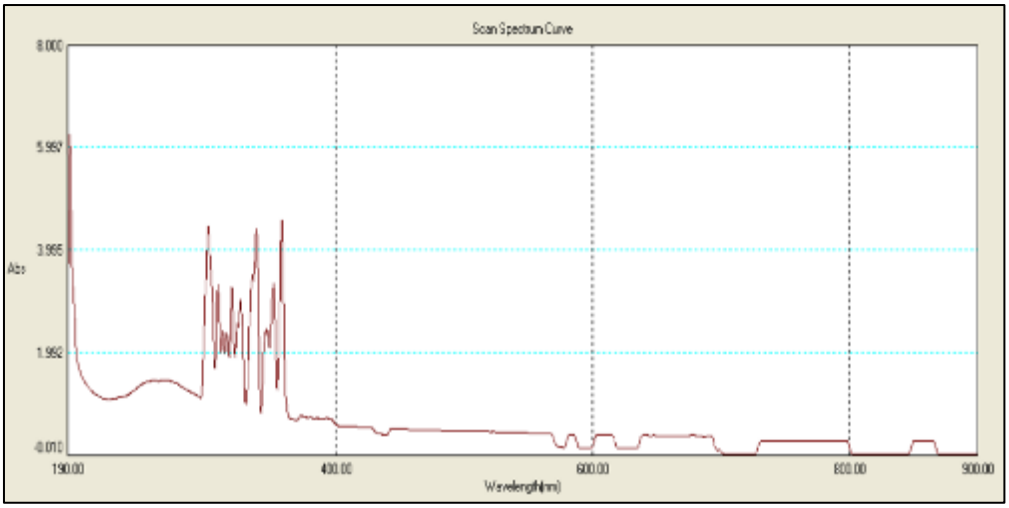
After the Completion of dispersion, we made five different samples from the dispersed SWCNTs solution and placed them in the sonication for 1 hour, 2 hours, 3 hours, 4 hours, and 5 hours with the control temperature as mentioned above. The prepared five samples of dispersed SWCNTs solution were centrifuged for 30 min at 1000 rpm, and a UV-visible spectrum was taken for each of the five samples, which we called a sample without dilution. 1 mL of each sample was taken after 30 min centrifugation and diluted with 1 mL of SDS solution by two times, respectively, and labeled as a sample of 2 times dilution. Similarly, 1 mL of SWCNTs solution was diluted by 1 mL of SDS solution by four times, respectively, and labeled as a sample of 4 times dilution. UV-visible spectrum was taken for each of the 10 diluted samples after each time the dilution sample was completed.

A total of fifteen samples were prepared with respect to dilution time and dilution factor Table S1. These fifteen samples were deposited onto the glass surface to prepare the samples for Surface morphology analysis by Dektak Vecco 150 Surface Profiler. The upper part of the glass surface was cleaned with ethanol properly to make sure there were no samples on that side, because of comparing the two different regions of the glass slide.

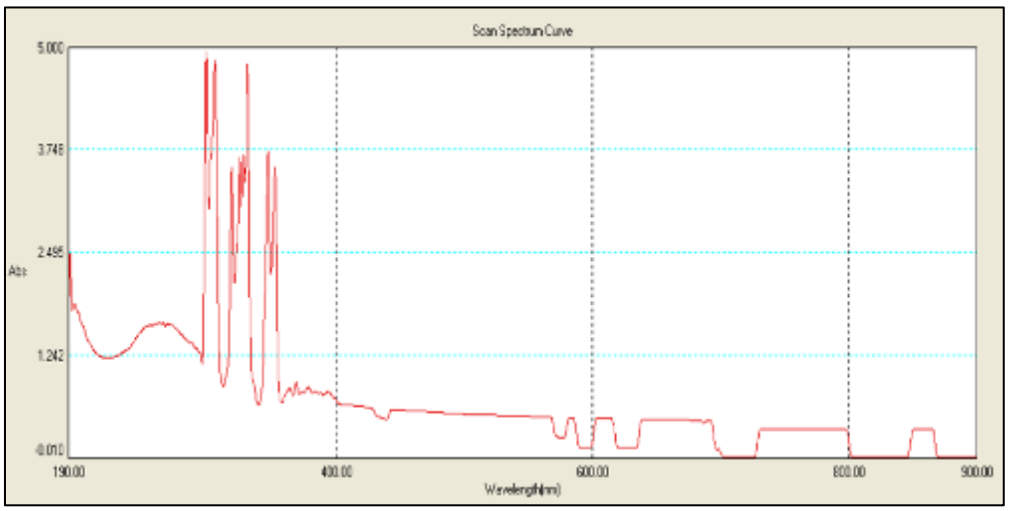
Five samples were selected for Scanning Electron Microscopy (SEM) Analysis from a total of fifteen samples. Those five samples were prepared by deposition on a Silicon wafer. Firstly, the Silicon wafer was cleaned with distilled water and ethanol in an Ultrasonic bath. Then the deposition was performed and dried normally, and to remove the solvent, samples were poured into DI water for 10 minutes and then dried at room temperature, and for stability, samples were heated at 100-150°C.

2.3. Characterization Technique

The fifteen Samples were characterized by UV-visible spectroscopy where we used the wavelength range from 190 nm to 900 nm for scanning the fifteen different samples shown in Figure S1, S2 and also in Figure 1.


Scan Spectrum Curve
8.000
5.997
3.995
1.992
-0.010
Abs
190.00
400.00
600.00
800.00
900.00
Wavelength(nm)

(a)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Abs
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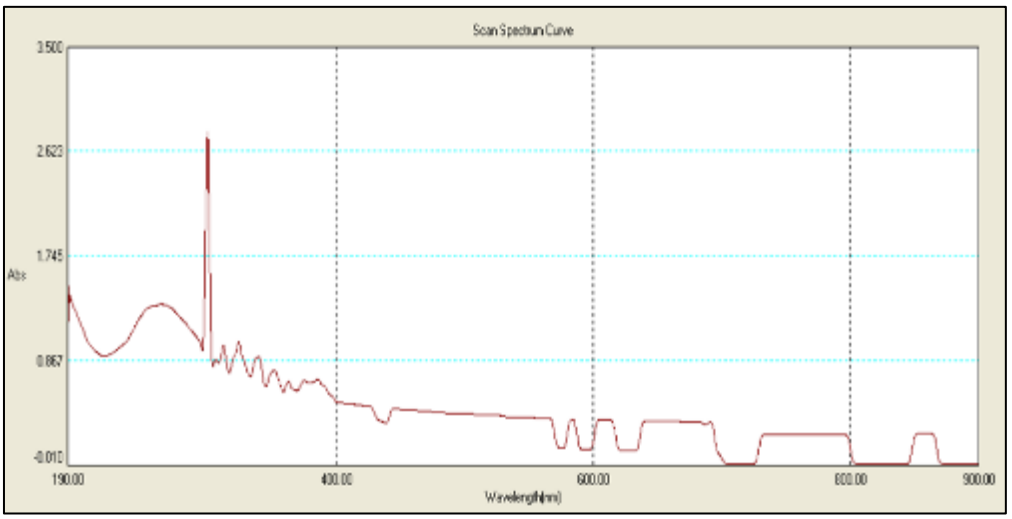
600.00
800.00
900.00
Wavelength(nm)

(b)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Abs
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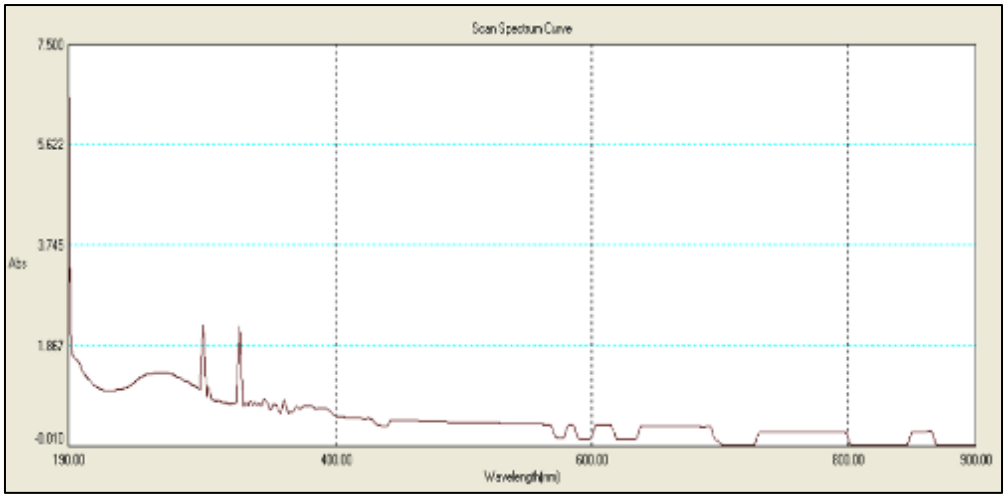
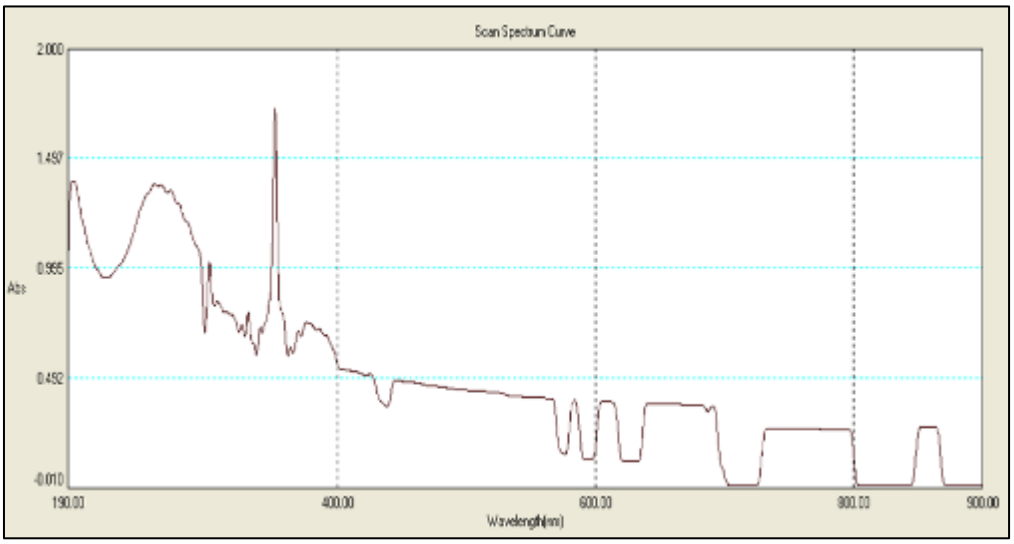
600.00
800.00
900.00
Wavelength(nm)

(c)



(d)



(e)

Figure 1 Five different UV-visible Spectra (a) S-1(4) (b) S-2(4) (c) S-3(4) (d) S-4(4) (e) S-5(4).

We also Dektak Vecco 150 Surface profiler to study the morphology of the prepared fifteen samples on the glass surfaces and the specifications are included in the supplementary information in Table S2, Table S3, and Table S4. Finally, the selected well-dispersed and uniformly distributed samples were studied by Scanning Electron Microscopy (SEM) to observe the homogenous distribution of nanotube formations on the deposited sample surface and also the surface pores of the selected samples.

3. Results and discussions

3.1. Study of Dispersion by UV-Visible Spectroscopy

Fifteen different UV-Visible spectra were taken to observe the dispersion properties of SWCNTs solution. The concentration of prepared SWCNTS solution was as follows more concentrated was without dilution, then 2 times dilution, then 4 times dilution. We observed significant differences in UV-Visible spectra among the fifteen samples according to dilution time and dilution factor, where the more concentrated sample, which was without dilution, did not show any significant absorbance on the UV-Visible spectrum among of the five without dilution samples Figure S1. The reason for the lower absorbance is that the debundling of SWCNT into the solution did not happen properly. However, the samples with 2 times dilution and 4 times dilution showed distinguishable absorbance on the UV-Visible spectra. Among the rest 10 samples of 2 times dilution and 4 times dilution, 2 times dilution samples showed the highest

absorbance Figure S2. However, each five of spectra showed similar absorbance patterns which were not clearly mentionable as well as cannot distinguish properly which sample debundle SWCNT into the solution properly. However, five samples of 4 times dilution showed clear distinguishable absorbance patterns on the UV-Visible spectra which can be observed in Figure 1. S-1(4) in Figure 1(a) and S-2(4) in Figure 1(b) showed the highest absorbance among the five 4 times dilutions samples in Figure 1 which indicated that the 4 times dilution along with 1 hour and 2 hours sonication was able to debundle the SWCNT into the solution properly though the concentration in 4 times dilution sample was lower than the other 10 samples.

3.2. Morphological Study of Dispersed Sample by Surface Profiler

In Figure S3(a), the most intense peak is for dust, and other peaks are so close and dense that's why it is not identifiable the CNT peak, but Figure S3(b) clearly shows, that the red line and green line compare two regions where the green line indicates the CNT peak area and From Figure 2, we observe that CNT peak are uniformly oriented all over the glass slide. In Figure S4(a), the red and green lines compare two regions but CNT peaks in the green region are so dense with dust peaks that cannot be distinguishable properly. Although from Figure S4(b), CNT peaks are identified but they are very insignificant. In Figure S4(c), CNT peaks are identified and compared in two regions by red line and green line and the CNT peaks are uniformly dispersed in green gone but some unusually intense peaks might be for dust particles. In Figure 3, the red line indicates there are no peak observed but green line indicated peaks are found and the orientation of them are uniform. From, Figure S5(a), cannot clarify the CNT peaks, but some are found which is for dust particles. The same results are observed in Figure S5(b) peaks are found all over the graph, but it is not clearly understood whether it is for CNT or for dust particles. It cannot be compared or clarified from Figure S6(a) neither CNT peaks nor dust peaks and also the peaks we found were not uniformly oriented. The same factors are observed in Figure S6(b) that the peaks we found from the two regions are neither it comparable nor it uniform and also dust peaks are observed which are the most intense. But we get some peaks from Figure 4 which are

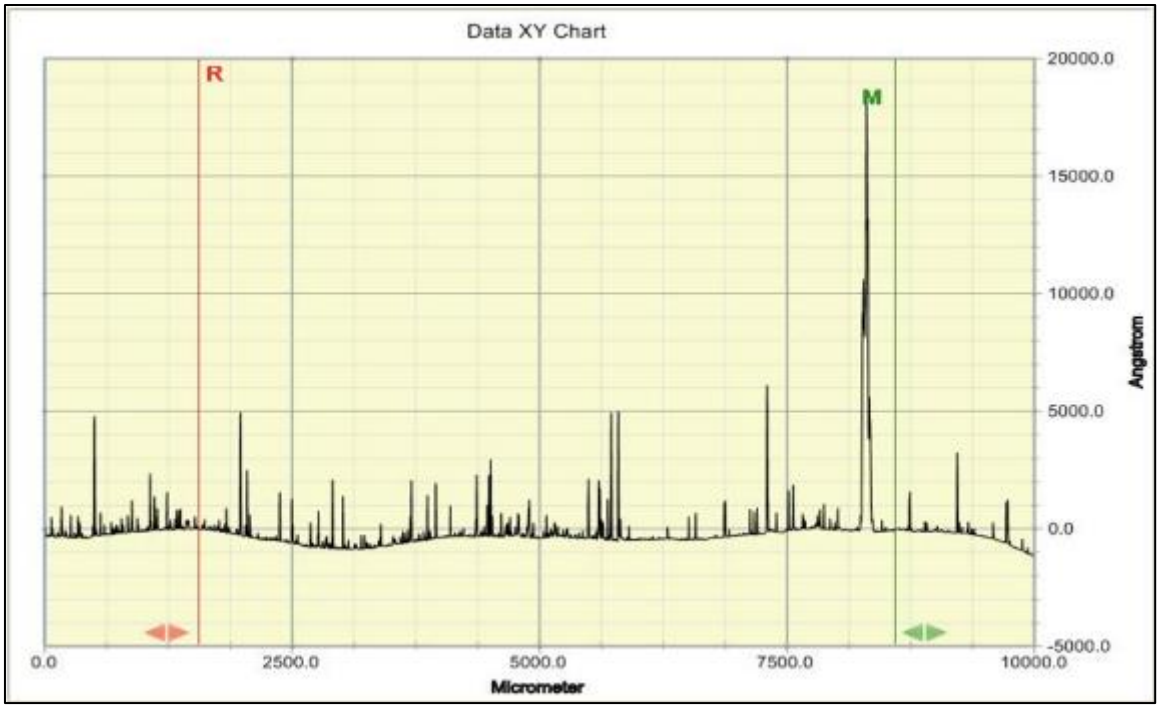


Figure 2 Surface Studied Samples for S-1(4 times dilution).

comparable and also uniformly oriented and also not all for dust particles. In Figure 5, it is clearly understood by two regions which is indicated by a red and green line that the peaks are identified in green oriented uniformly and also not only for dust peaks but also for CNT peaks. Dust peaks are the most intense than CNT peaks.

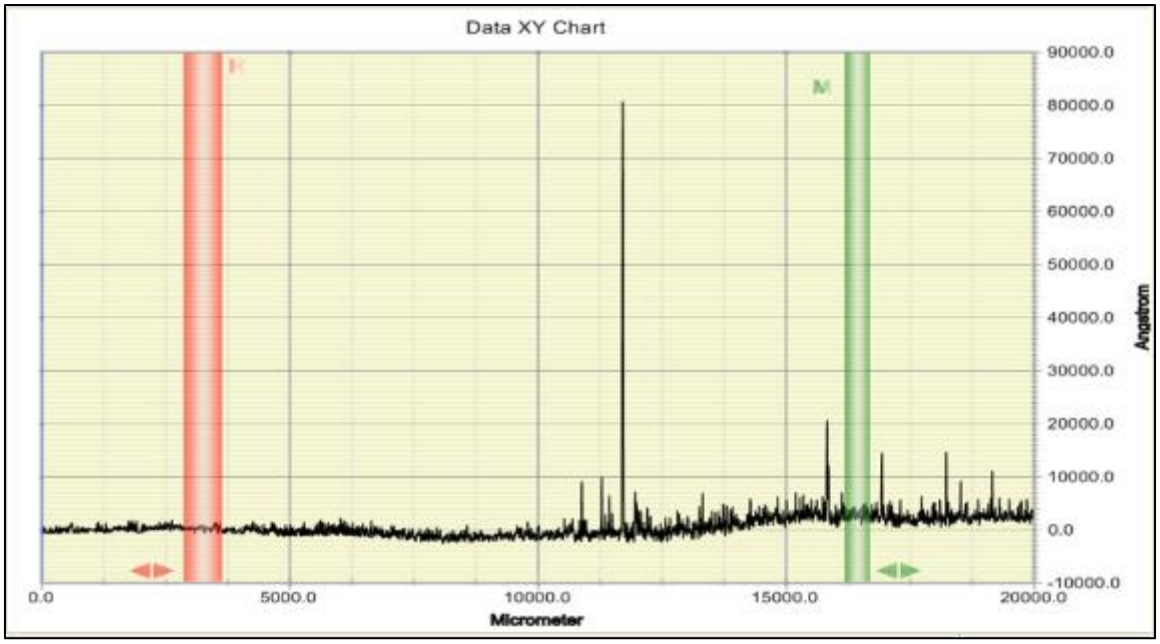


Figure 3 Surface Studied Sample for S-3 (without dilution).

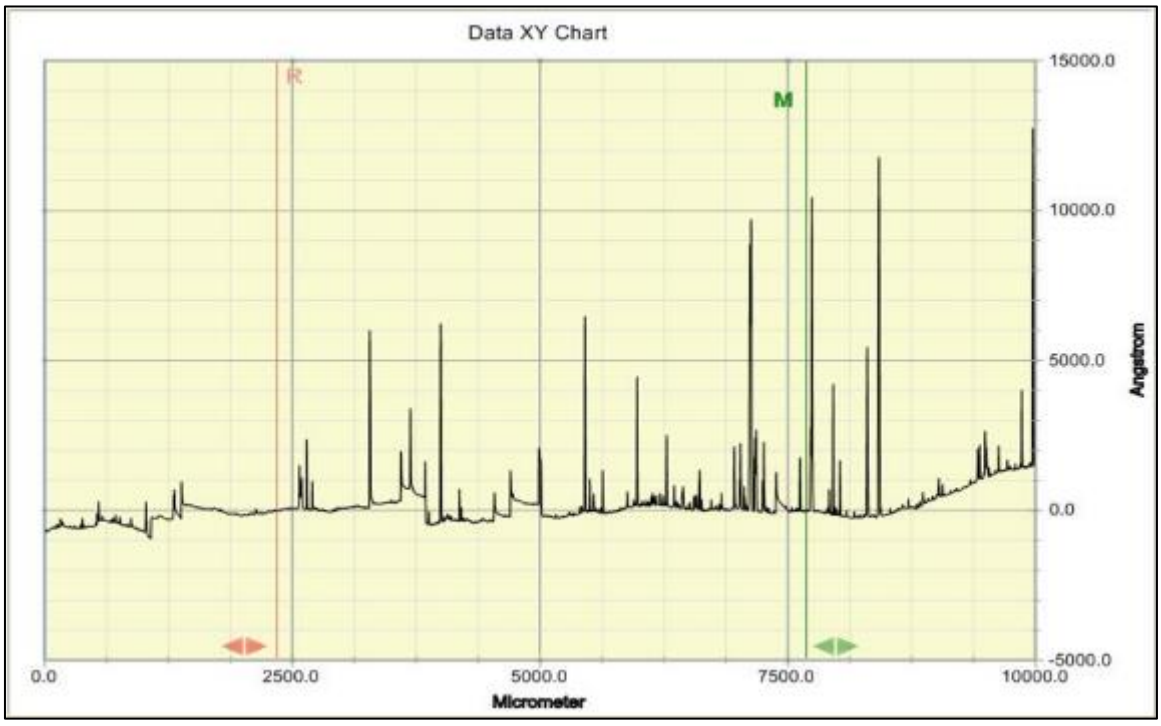


Figure 4 Surface Studied Sample for S-4 (4 times dilution).

However, the peaks we observe all over the graph from Figure S7(a) and S7(b) are not clearly understood and cannot be identified whether for dust or CNT peaks. Among the fifteen samples, we observed that sample-1(4 times dilution), Sample-3(without dilution), Sample-4(4 times dilution), and Sample-5 (without dilutions) are well dispersed and uniformly distributed.

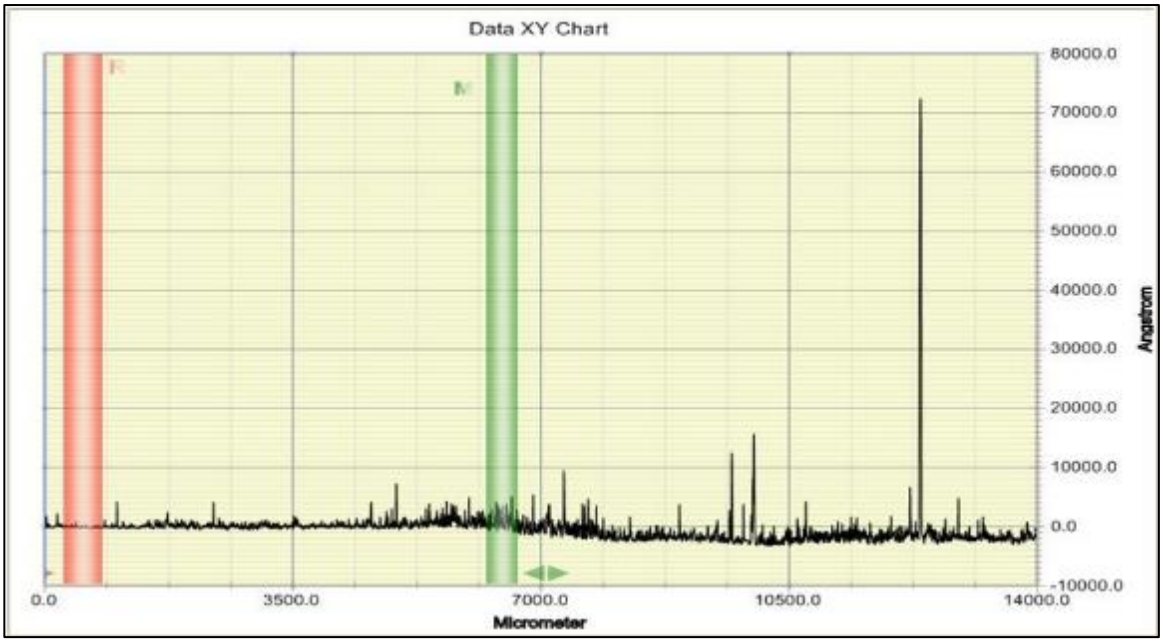
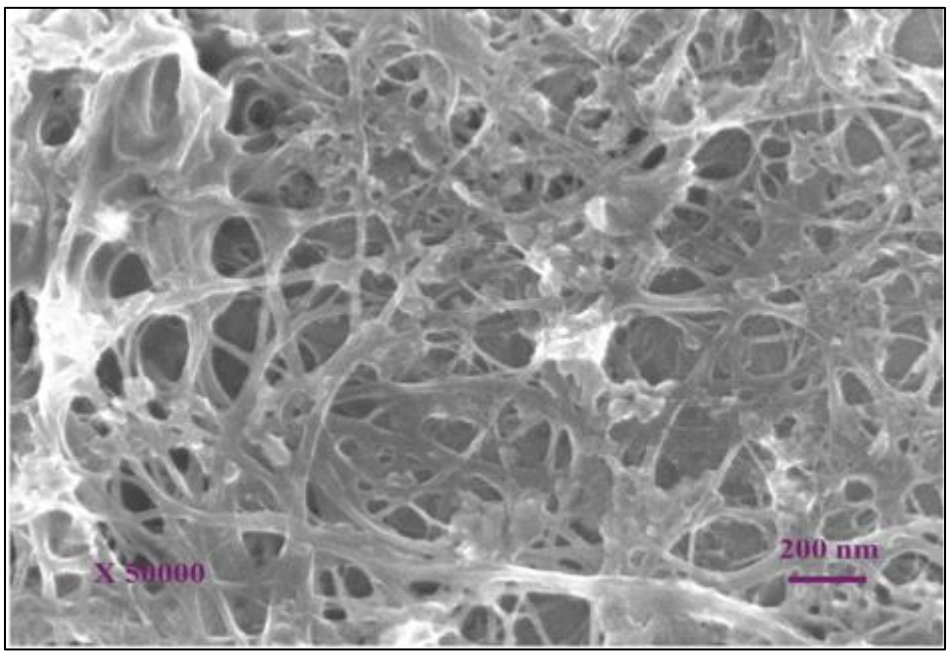


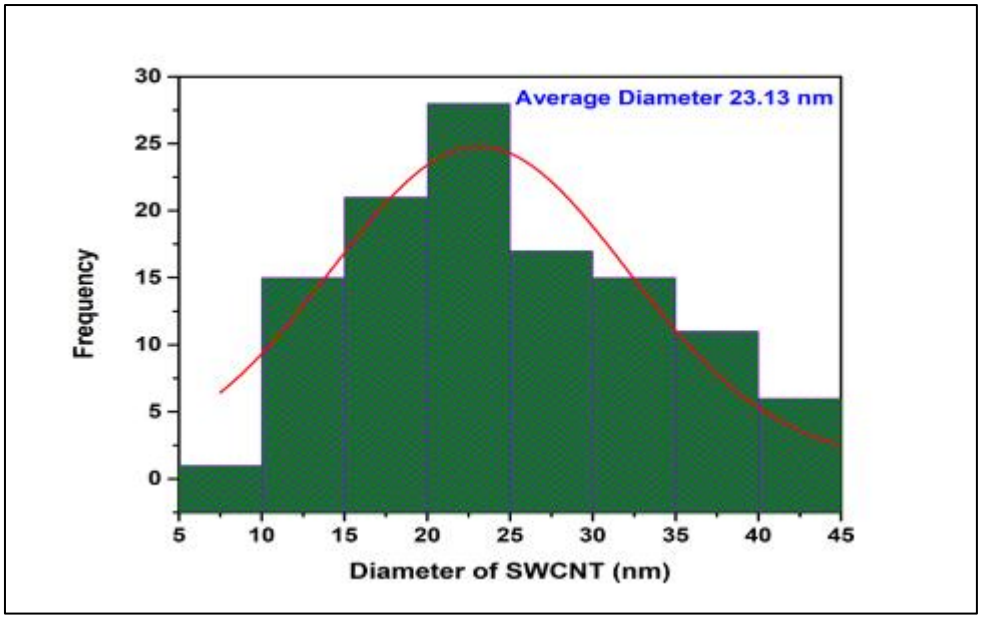
Figure 5 Surface Studied Sample for S-5 (without dilution).

3.3. Study of Surface Property by Scanning Electron Microscopy (SEM)

SEM analysis of sample-1 (4 times dilution) in Figure 6(a) showed that the pores we get by debundling of nanotubes are uniformly distributed and the diameter of the pores is averagely the same and also nanotubes are not bundled.



(a)



(b)

Figure 6 SEM Images for Sample S-1(4) (a) Overall Surface (b) Diameter Distribution Profile of SWCNT

Nanotubes are uniformly oriented all over the place, but they are so dense because of the concentration of the solution. Figure 6(b) shows the distribution profile of the SWCNTs. Sample-3 (without Dilution) in Figure 7 showed that nanotubes are not dispersed uniformly, and they are bundled in one place.

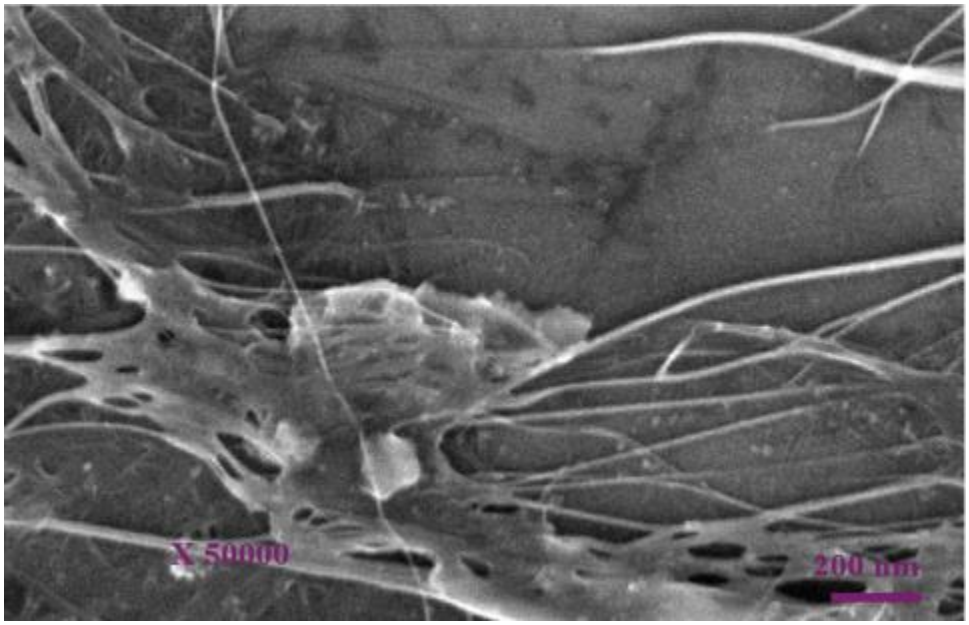


Figure 7 SEM Image for Sample S-3.

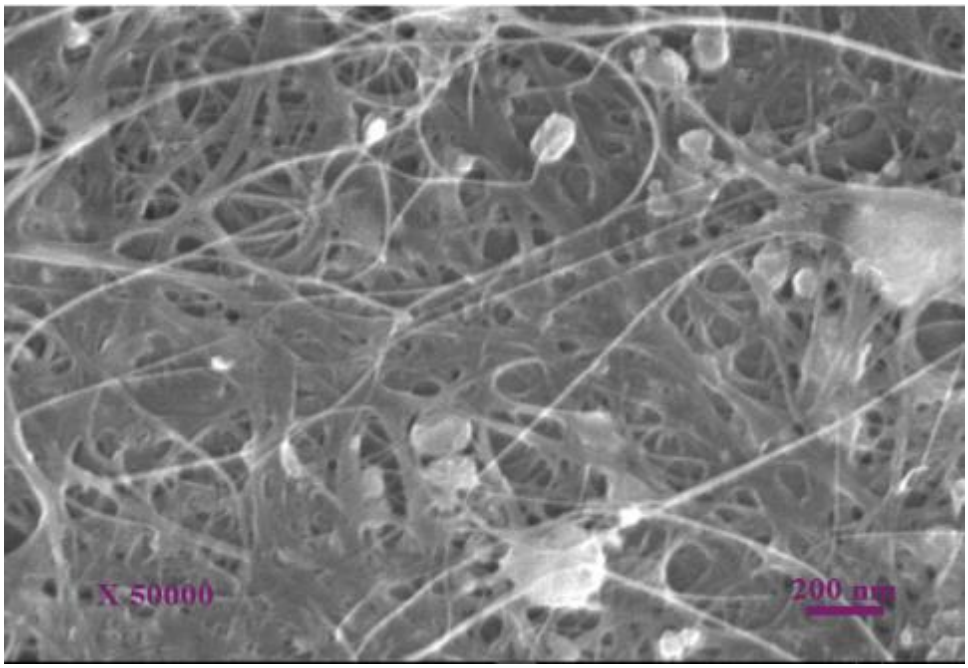


Figure 8 SEM Image for Sample S-4(4).

Sample-4 (4 times dilution) in Figure 8 Shows that nanotubes are not debundled properly, although the pores of the figure and length of nanotubes indicate that CNT is uniformly oriented.

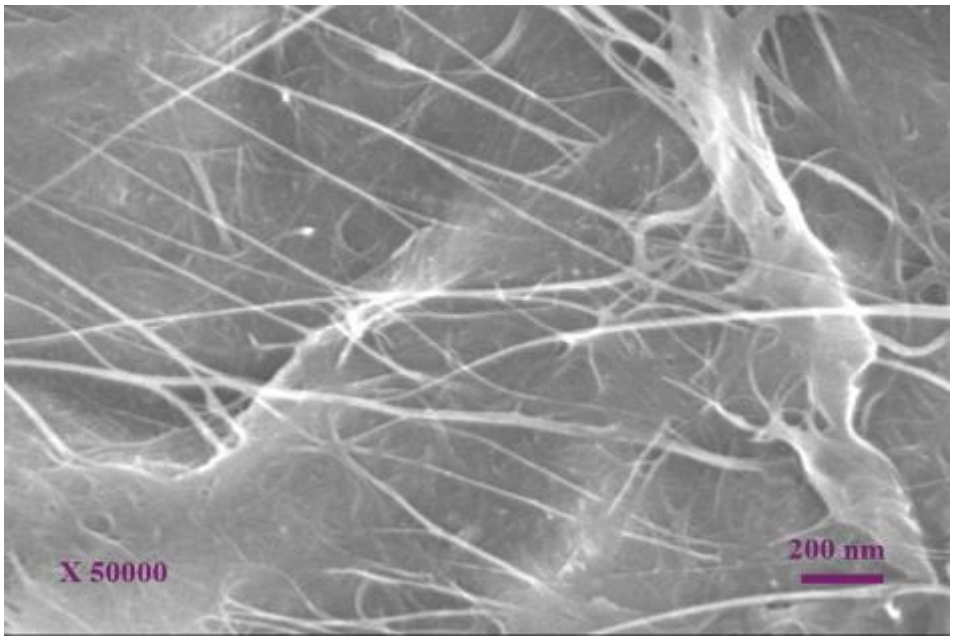


Figure 9 SEM Image for Sample S-5.

Finally, Sample-5 (without dilution) in Figure 9 clearly shows that CNTs are not dispersed well enough to distribute uniformly, though some nanotube structures are observable, but neither dispersed properly nor uniformly distributed. The Surface study shows us that Sample-1 (4 times dilution) is a well-dispersed and uniformly distributed sample among the fifteen samples.

4. Conclusions

we report a simple UV-Visible spectroscopic and SEM technique to monitor the sonication-driven dispersion of SWCNTs in aqueous SDS solutions. The results show that the maximum achievable dispersion corresponds to the maximum UV absorbance of the SWCNTs solution. Moreover, we have demonstrated moderately uniform dispersion of SWCNTs in aqueous SDS solutions. The sonication-driven dispersion and the factors that optimize its effect have also been systematically studied by using the Surface profilometer and SEM. SWCNTs are gradually disentangled from aggregates and bundles and stabilized by SDS. The SDS molecules are adsorbed on the surface of SWCNTs and prevent re-aggregation. The moderate dispersion of the SWCNTs is 0.3mg; the optimal concentration of SDS is 1.5 wt%; low temperature (24°C), and the appropriate increase in sonication time contribute to the dispersion of carbon nanotubes. However, this work could be an application of Solar-Cell System, Gas Sensor, and other renewable energy technologies.

Compliance with ethical standard

Disclosure of Conflicts of Interest

We, all the authors, hereby declare that there are no conflicts of interest.

Funding

This work is supported by no external funding.

Supporting Information

The Data for supporting information is available.

Author Contributions:

- MD Ruhul Amin: Conceptualization, methodology, data curation, Validation, Investigation, writing- original draft.
- Md. Mosharraf Hossain Bhuiyan: Project Administration, conceptualization, Supervision, writing-review and editing.
- Monirul Islam Uzzal: data curation, formal analysis, visualization, resources.
- Fumiaki Mitsugi: visualization, resources, software.

Data Availability Statement

Data will be made available by the corresponding author upon reasonable request.

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Supplementary Information

Table S1 Sampling Label of fifteen different sample

		Sonication Time (hours)				
		1	**2**	**3**	**4**	**5**
Dilution Factors	**Without dilution**	S-1	S-2	S-3	S-4	S-5
	2-time dilution	S-1(2)	S-2(2)	S-3(2)	S-4(2)	S-5(2)
	4-time dilution	S-1(4)	S-2(4)	S-3(4)	S-4(4)	S-5(4)

The Sampling Label was created in Table S1 according to sonication time and dilution factors where, S = Sample, 1 to 5 = Sonication hours, and 2,4 under the first bracket = dilution factors.

Table S2 Scan parameters for five different samples of without dilution with different sonication times.

Sample	Scan type	Stylus Radius(μm)	Length (um)	Duration (sec)	Resolution (um/sample)	Force(mg)	Measure range(um)	Profile
S-1	Standard scan	12.5	15000.0	70	0.714	1.00	65.5	Hills&valleys
S-2	Standard scan	12.5	20000.0	80	0.833	1.00	65.5	Hills&valleys
S-3	Standard scan	12.5	20000.0	80	0.833	1.00	524	Hills&valleys
S-4	Standard scan	12.5	12000.0	80	0.500	1.50	65.5	Hills&valleys
S-5	Standard scan	12.5	14000.0	90	0.519	1.50	65.5	Hills&valleys

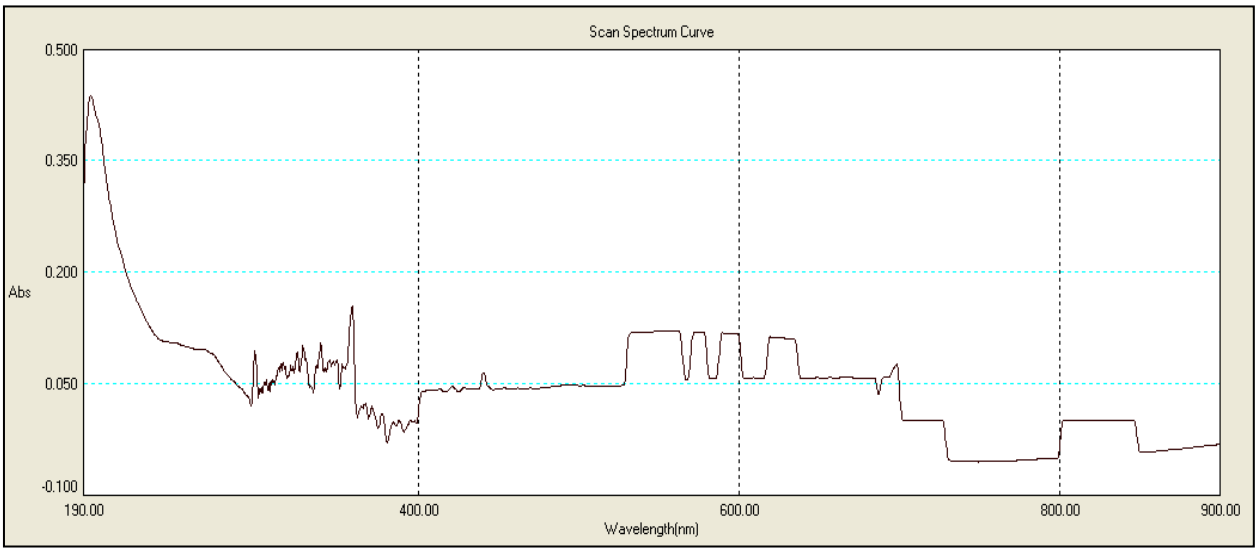
Table S3 Scan parameters for five different samples of 2-time dilution with different sonication times.

Sample	Scan type	Stylus radius(μm)	Length (um)	Duration (sec)	Resolution (um/sample)	Force (mg)	Measure range(um)	Profile
S-1(2)	Standard scan	12.5	11000.0	90	0.407	1.50	65.5	Hills&valleys
S-2(2)	Standard scan	12.5	11000.0	90	0.407	1.50	65.5	Hills&valleys
S-3(2)	Standard scan	12.5	11000.0	90	0.407	1.50	65.5	Hills&valleys
S-4(2)	Standard scan	12.5	11000.0	90	0.407	1.50	65.5	Hills&valleys

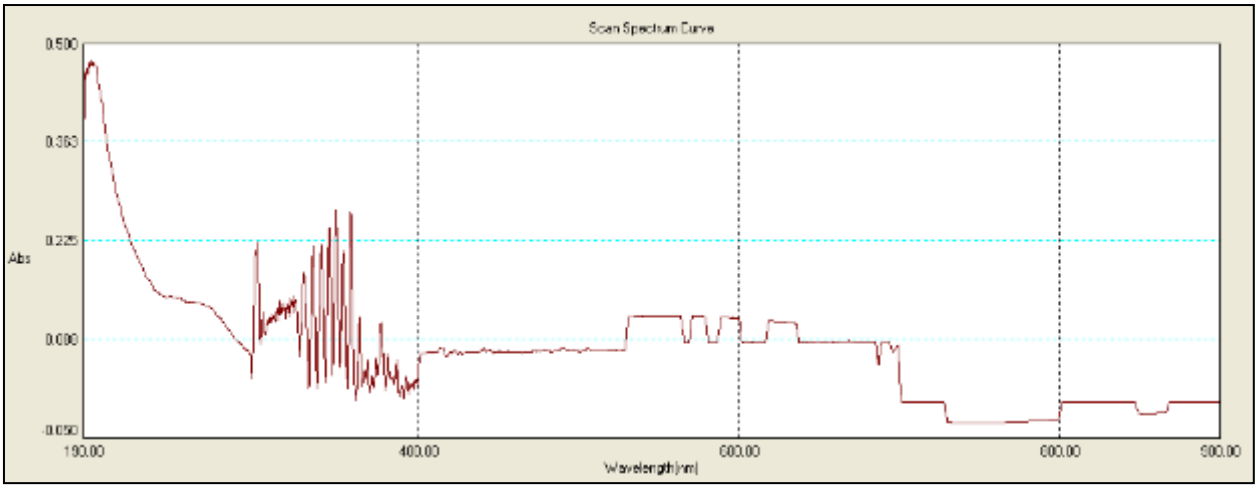
S-5(2)	Standard scan	12.5	15000.0	90	0.556	1.50	65.5	Hills&valleys

Table S4 Scan parameters for five different samples of 4-time dilution with different sonication times.

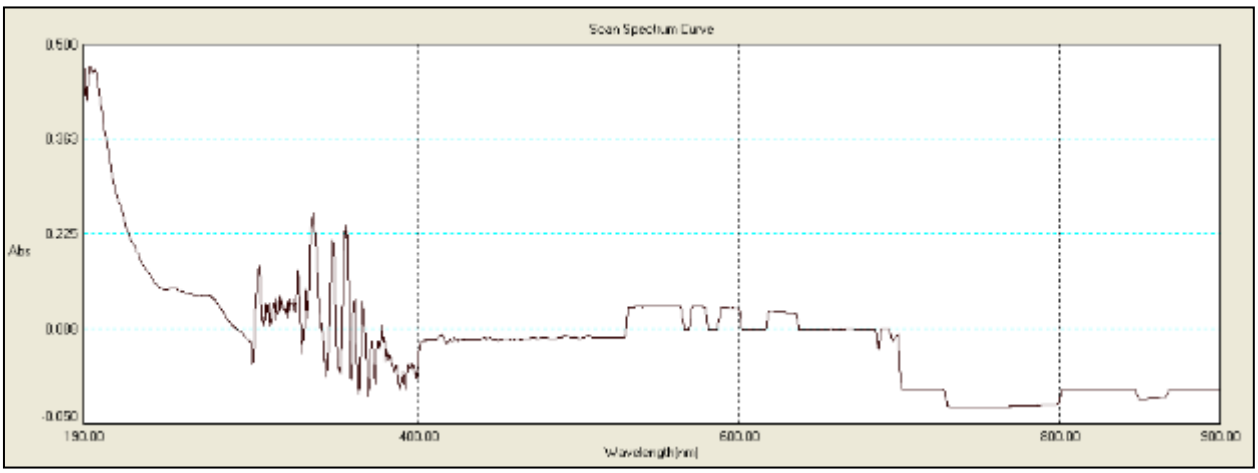
Sample	Scan type	Stylus radius(µm)	Length (um)	Duration (Sec)	Resolution (um/sample)	Force (mg)	Measure range(um)	Profile
S-1(4)	Standard Scan	12.5	10000.0	70	0.476	1.00	65.5	Hills&valleys
S-2(4)	Standard Scan	12.5	10000.0	70	0.476	1.00	65.5	Hills&valleys
S-3(4)	Standard Scan	12.5	10000.0	70	0.476	1.00	65.5	Hills&valleys
S-4(4)	Standard Scan	12.5	10000.0	70	0.476	1.00	65.5	Hills&valleys
S-5(4)	Standard Scan	12.5	10000.0	70	0.476	1.00	65.5	Hills&valleys



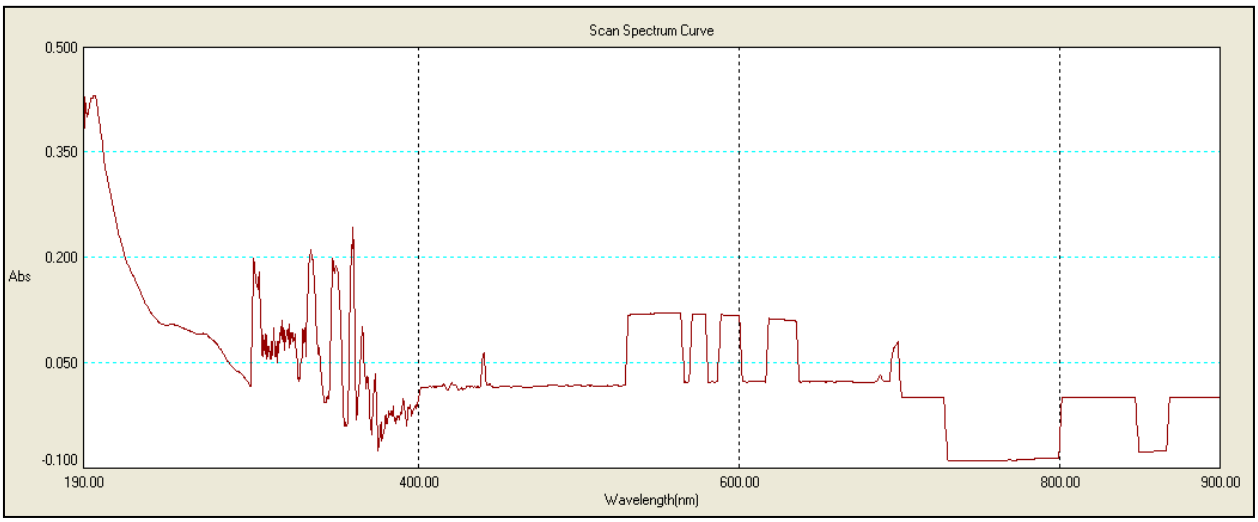
(a)


Scan Spectrum Curve
0.500
0.363
0.225
0.088
-0.050
Abs
190.00
400.00
600.00
800.00
900.00
Wavelength(nm)

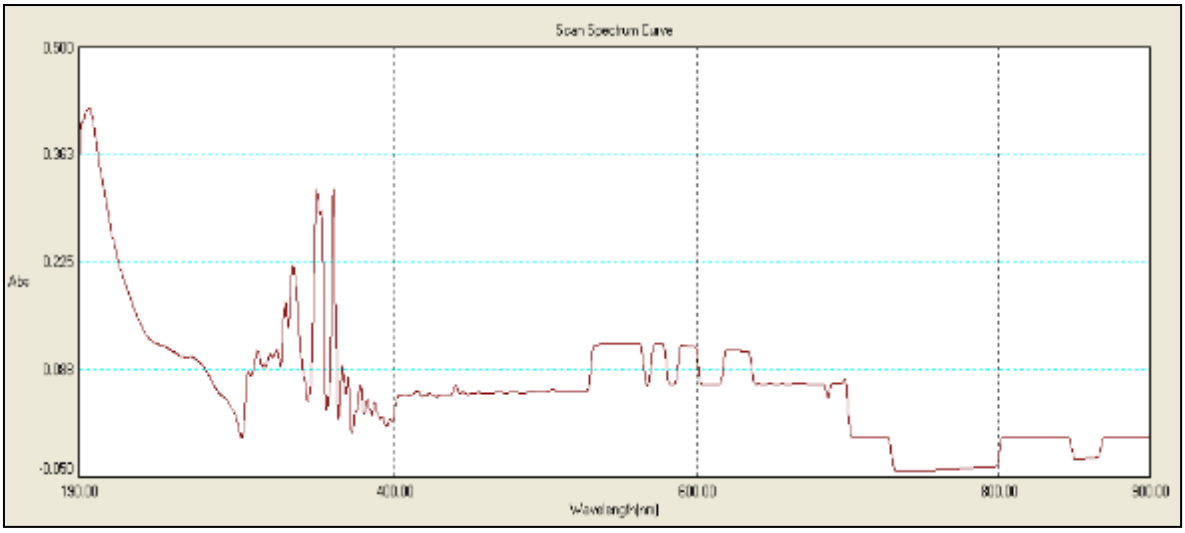
(b)


Scan Spectrum Curve
0.500
0.363
0.225
0.088
-0.050
Abs
190.00
400.00
600.00
800.00
900.00
Wavelength(nm)

(c)

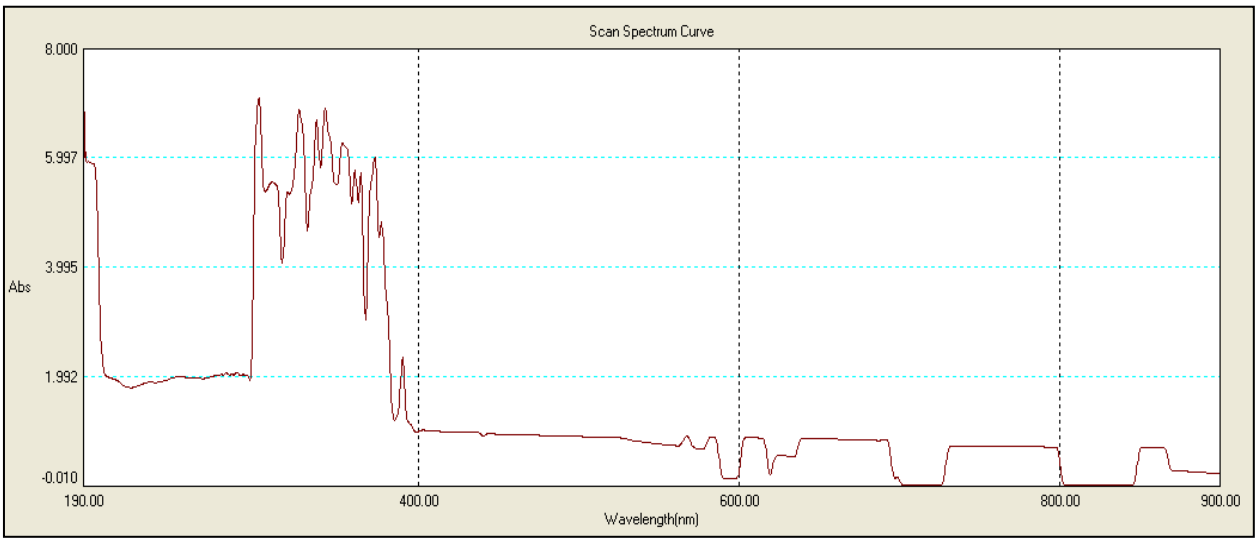

Scan Spectrum Curve
0.500
0.350
0.200
0.050
-0.100
Abs
190.00
400.00
600.00
800.00
900.00
Wavelength(nm)

(d)

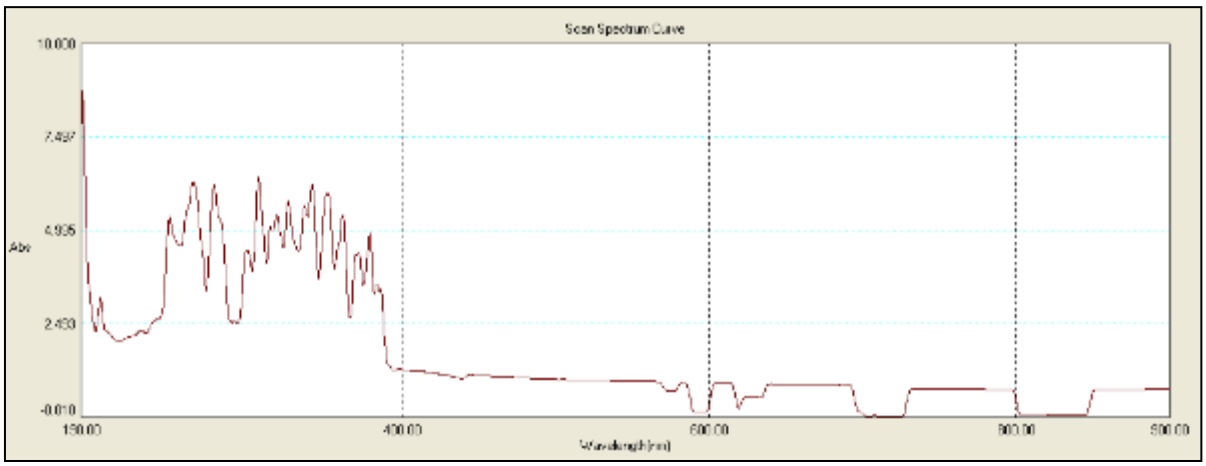


(e)

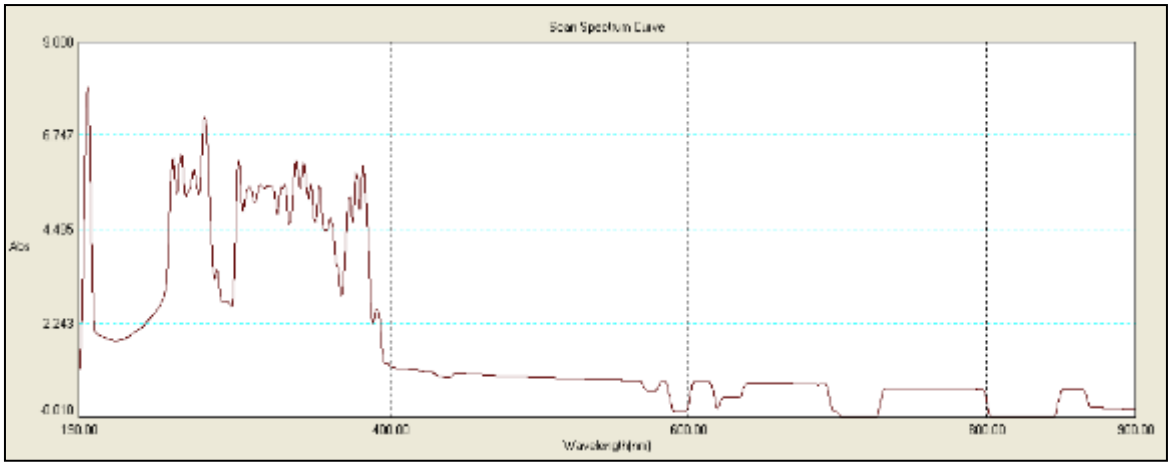
Figure S1 UV-visible Spectrum of five different samples without dilution where (a) S-1 (b) S-2 (c) S-3 (d) S-4 (e) S-5



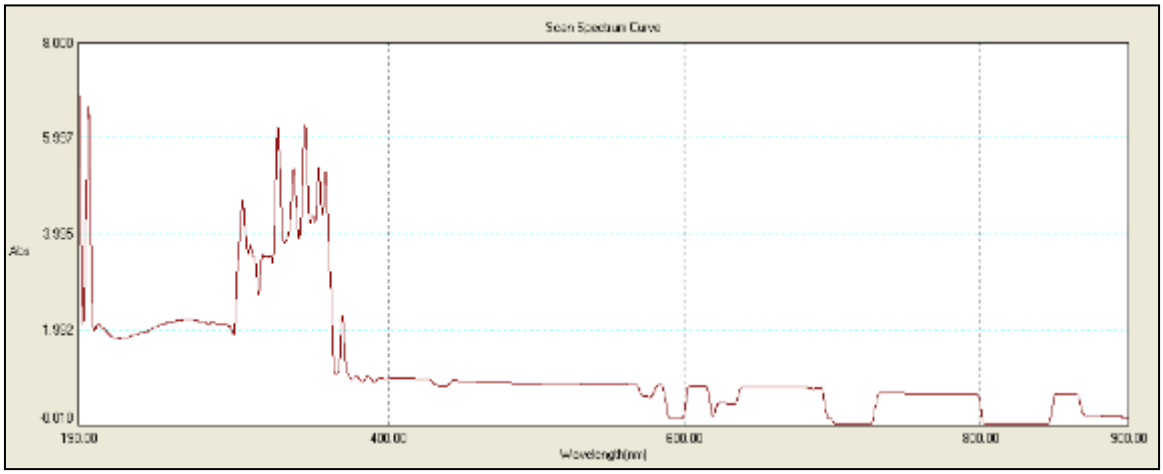
(a)



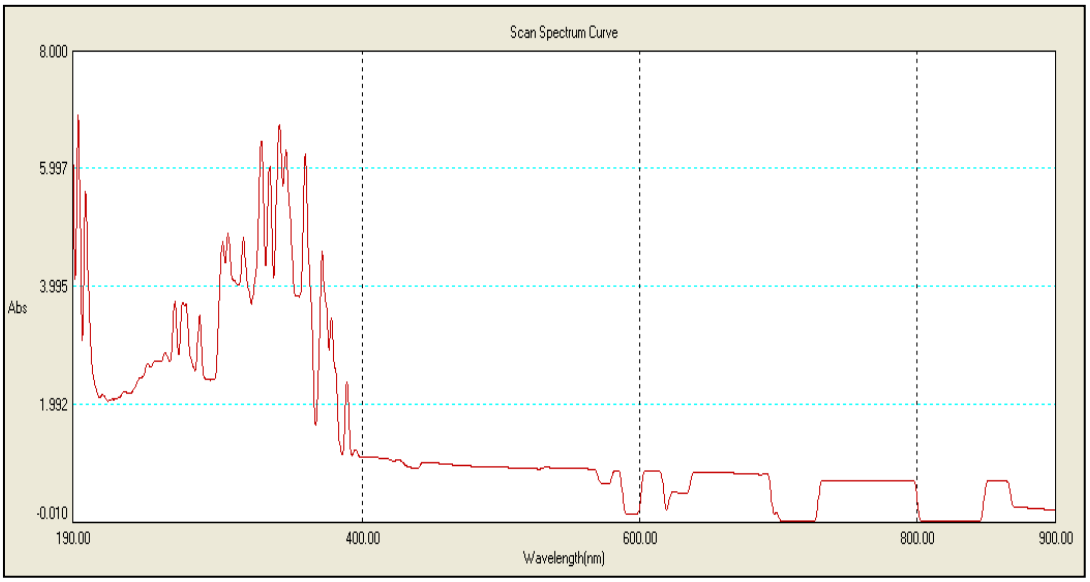
(b)



(c)

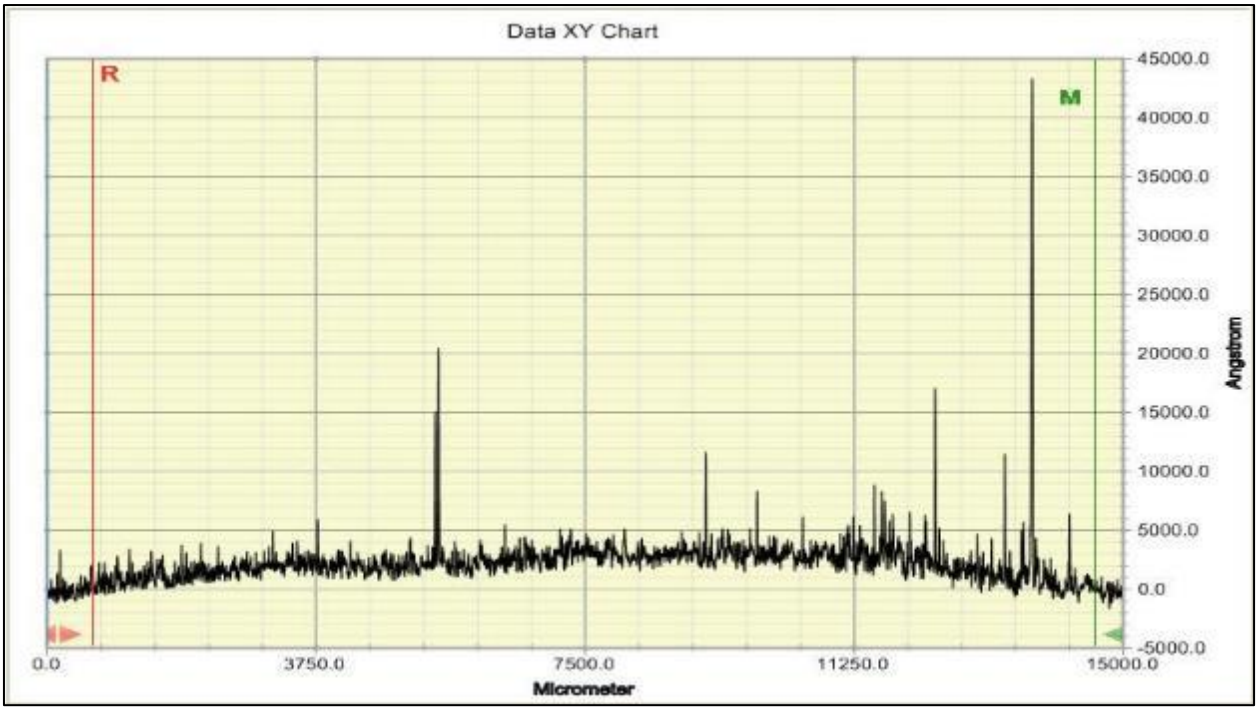


(d)

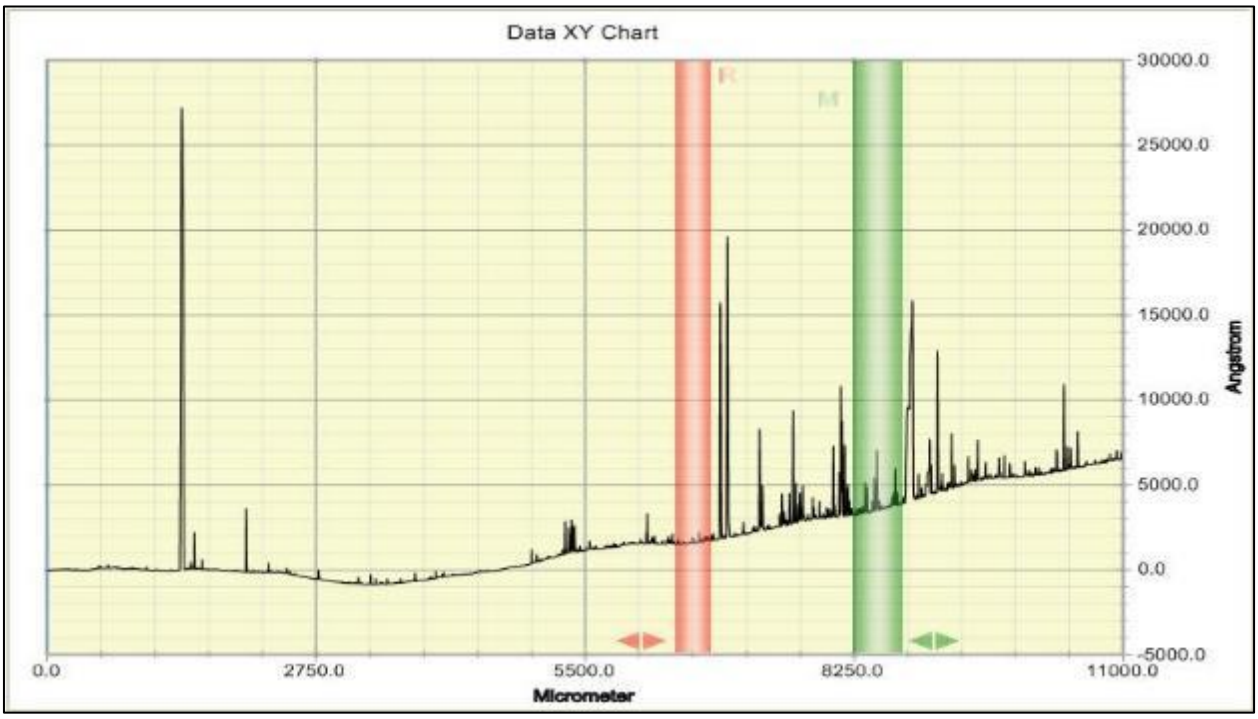


(e)

Figure S2 UV-visible Spectrum of five different samples of 2-time dilution where (a) S-1(2) (b) S-2(2) (c) S-3(2) (d) S-4(2) (e) S-5(2).

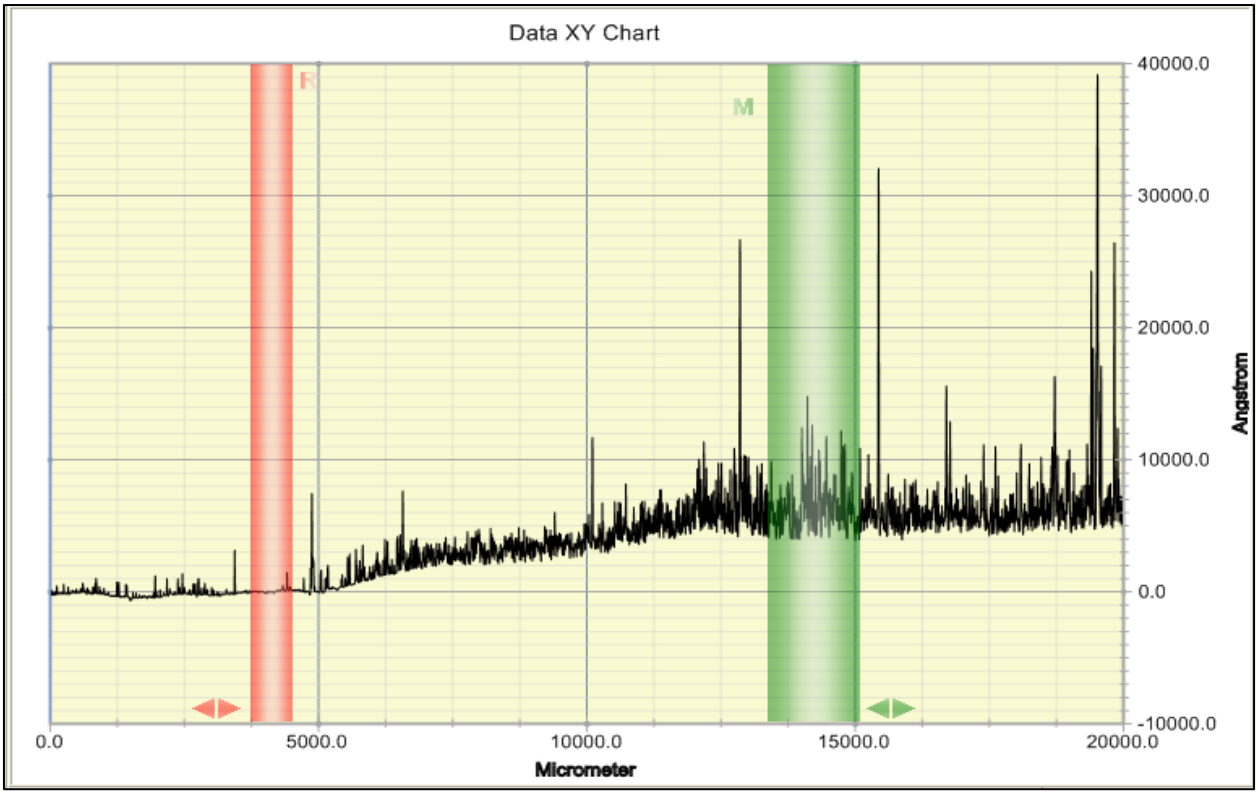


(a)

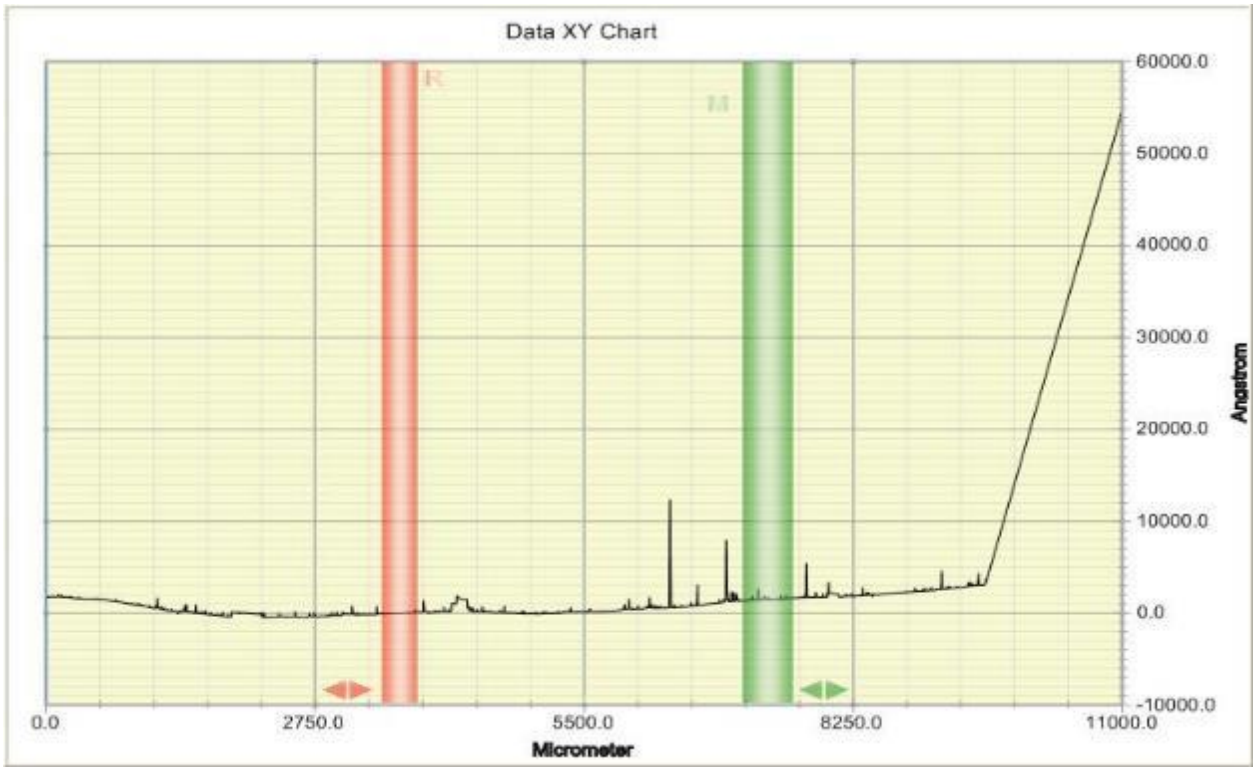


(b)

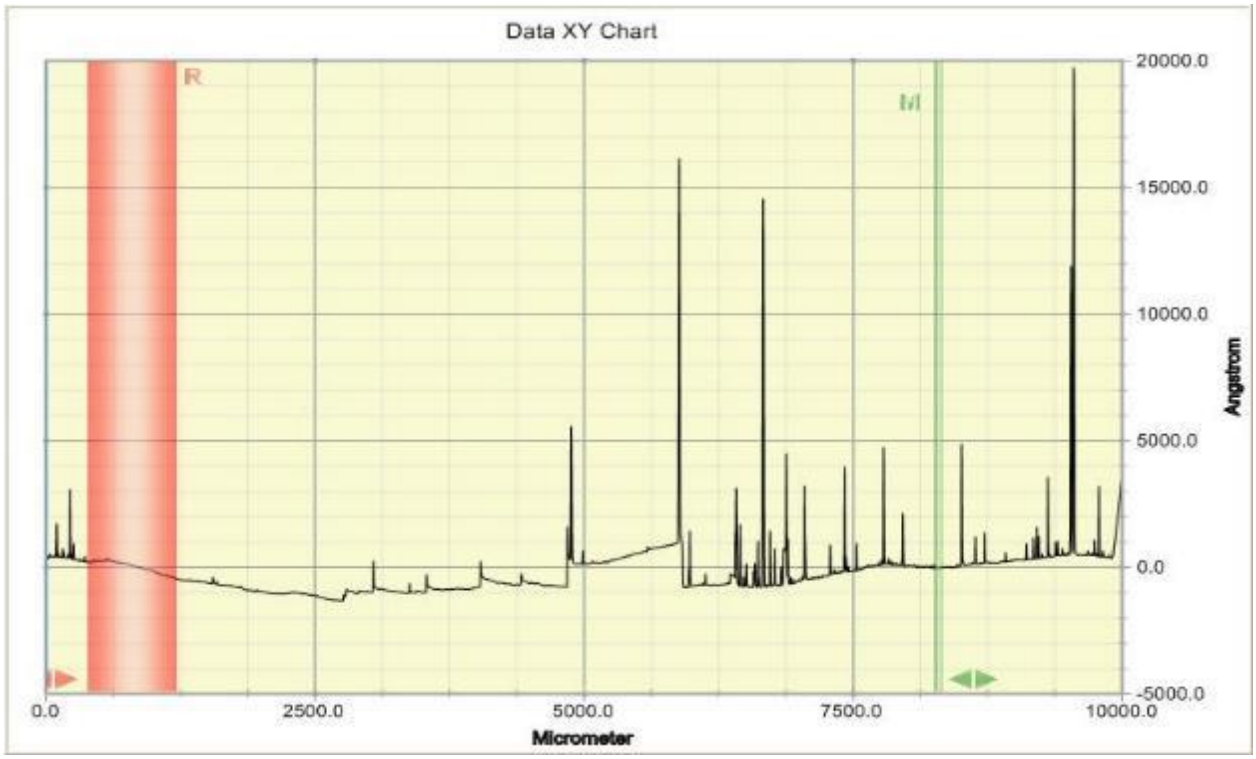
Figure S3 Two different Surface Studied Samples (a) S-1 (b) S-1(2).


Data XY Chart
R
M
40000.0
30000.0
20000.0
10000.0
0.0
-10000.0
Angstrom
0.0
5000.0
10000.0
15000.0
20000.0
Micrometer

(a)

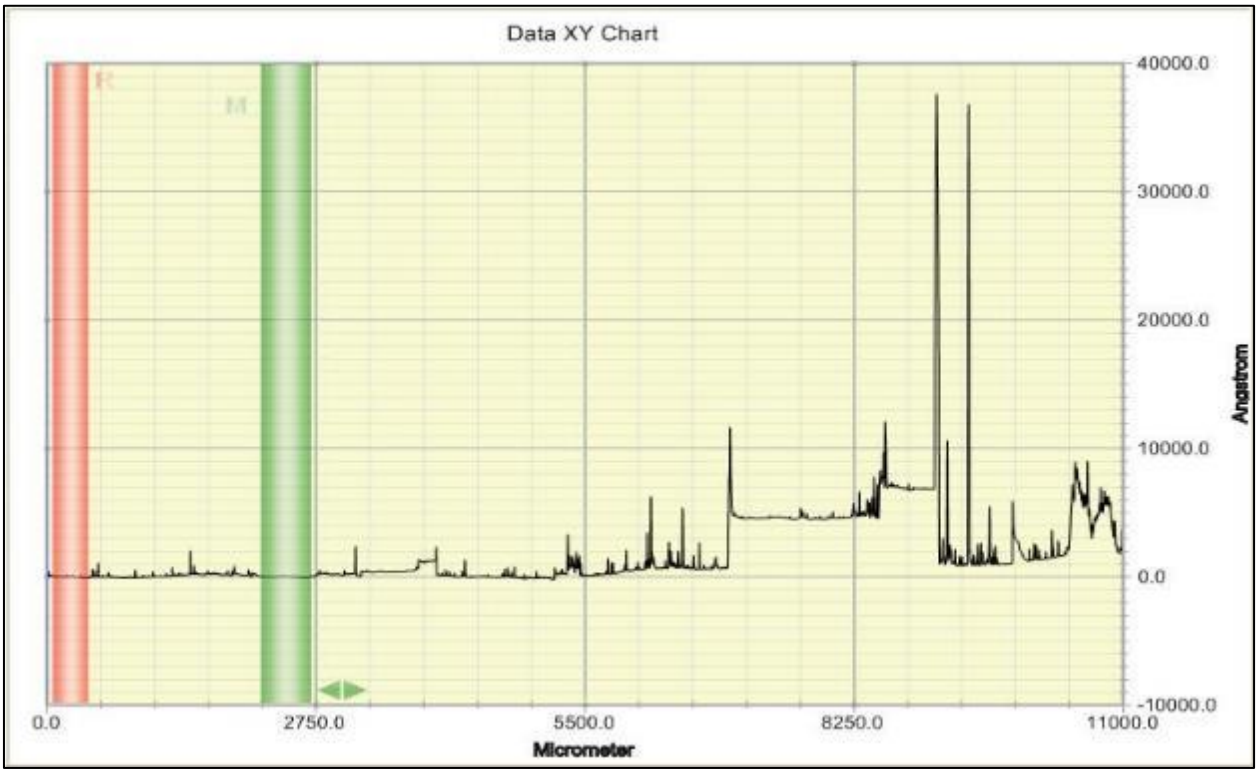

Data XY Chart
R
M
60000.0
50000.0
40000.0
30000.0
20000.0
10000.0
0.0
-10000.0
Angstrom
0.0
2750.0
5500.0
8250.0
11000.0
Micrometer

(b)

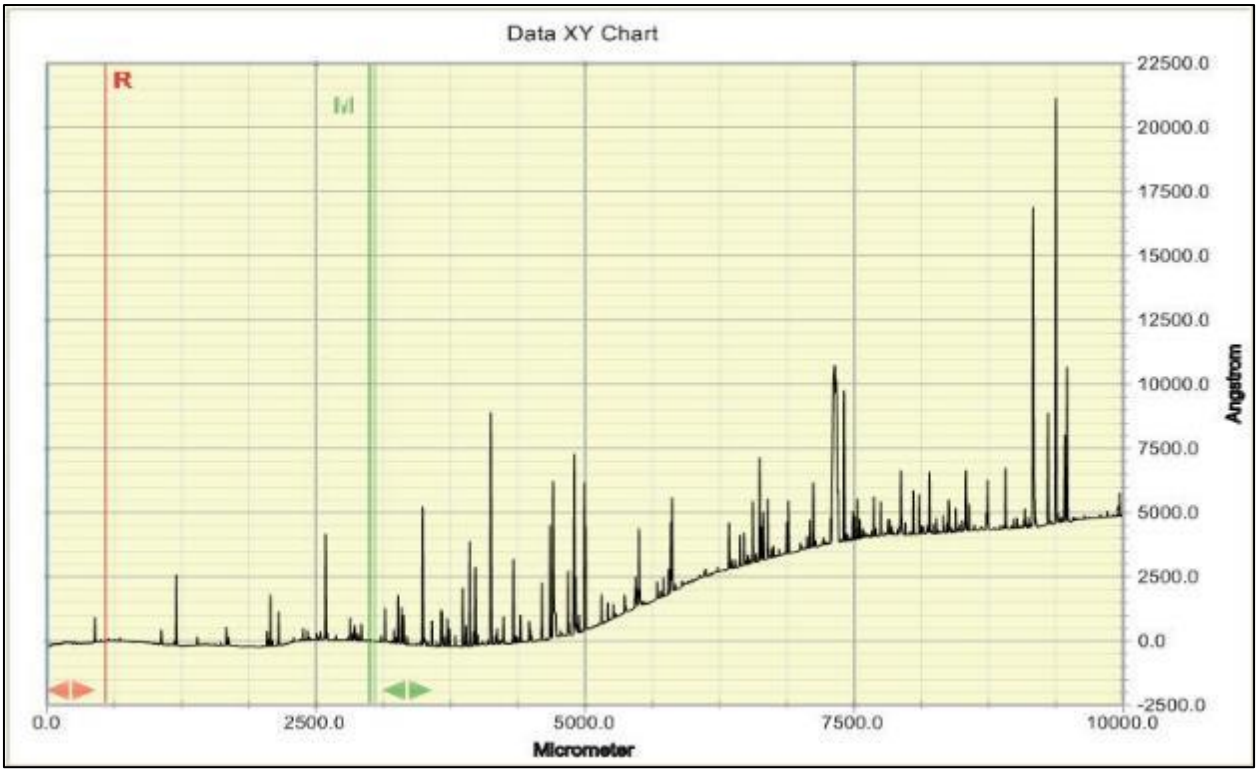


(c)

Figure S4 Three different Surface Studied Samples (a) S-2 (b) S-2(2) (c) S-2(4).

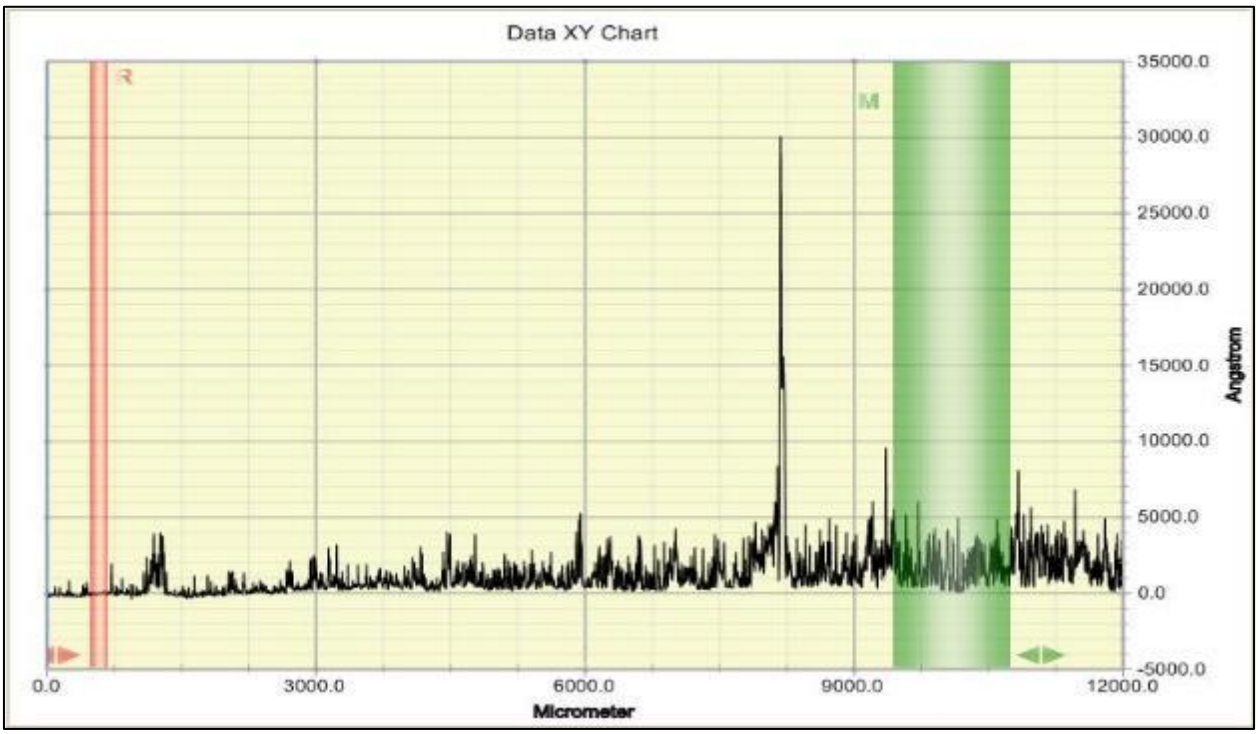


(a)

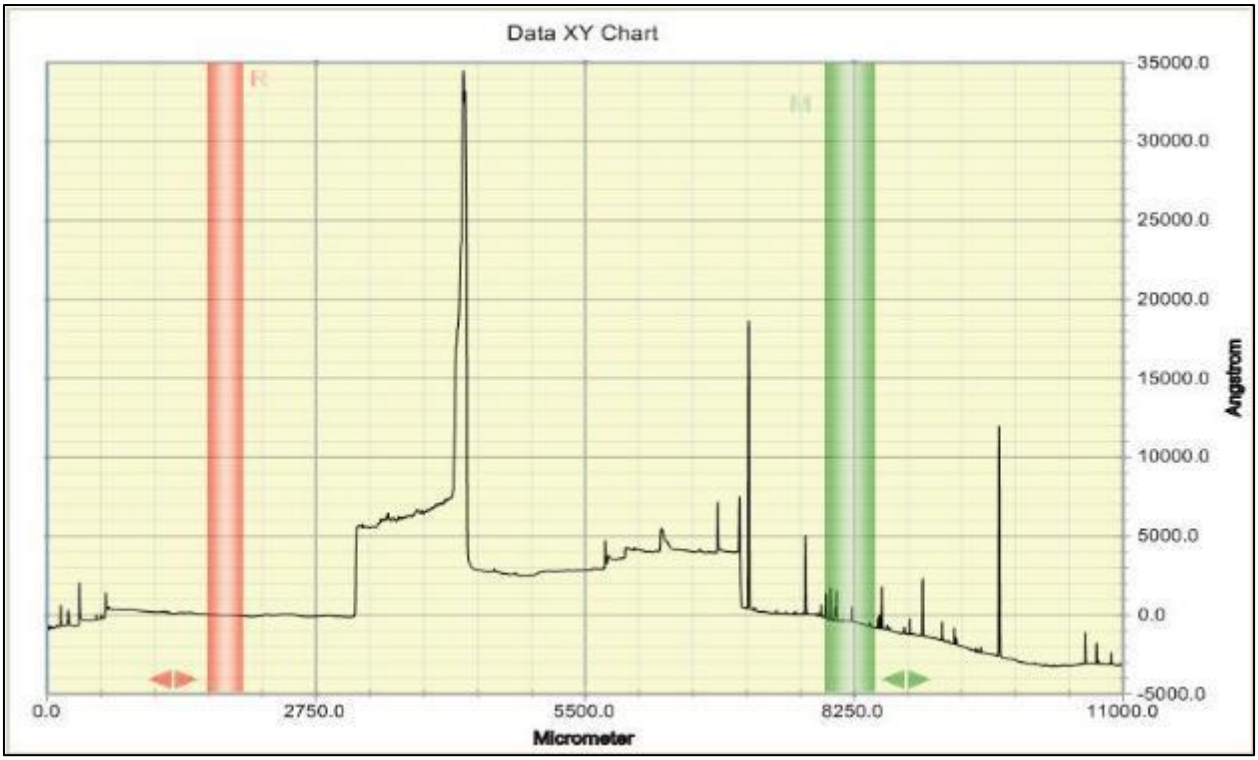


(b)

Figure S5 Two different Surface Studied Samples (a) S-3(2) (b) S-3(4).

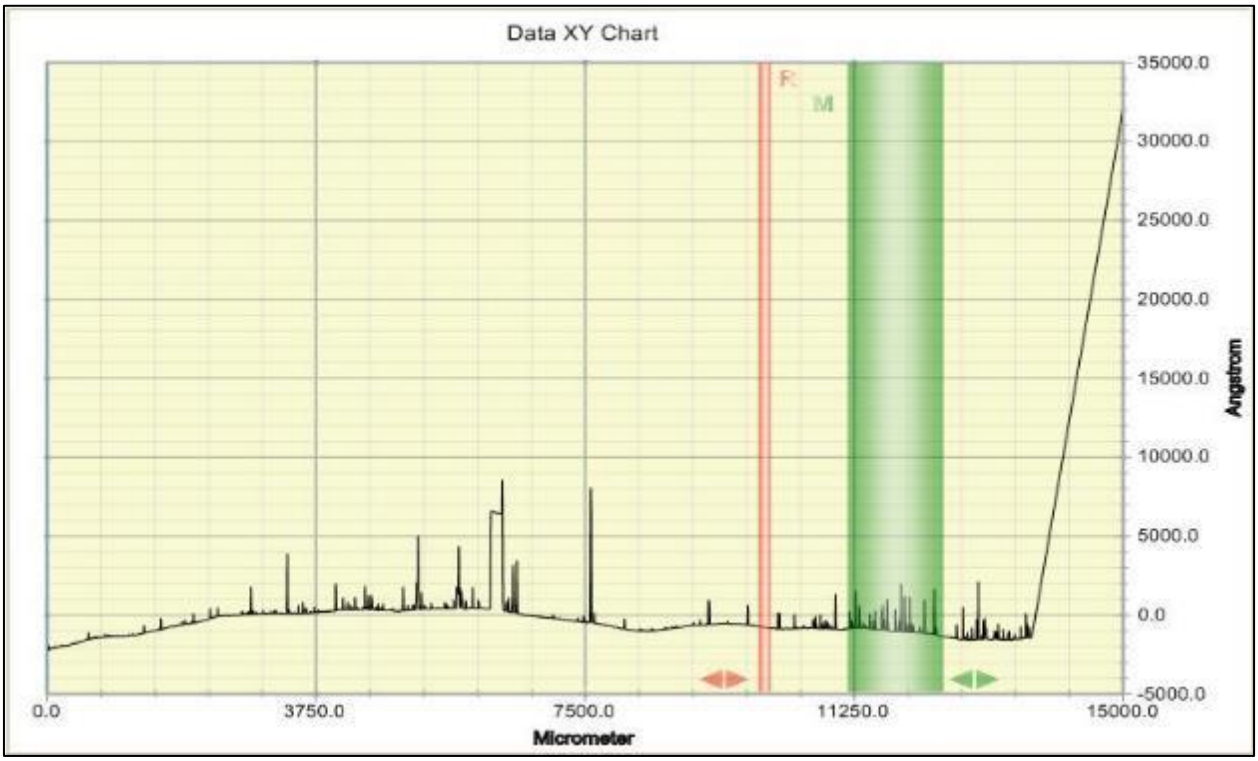


(a)

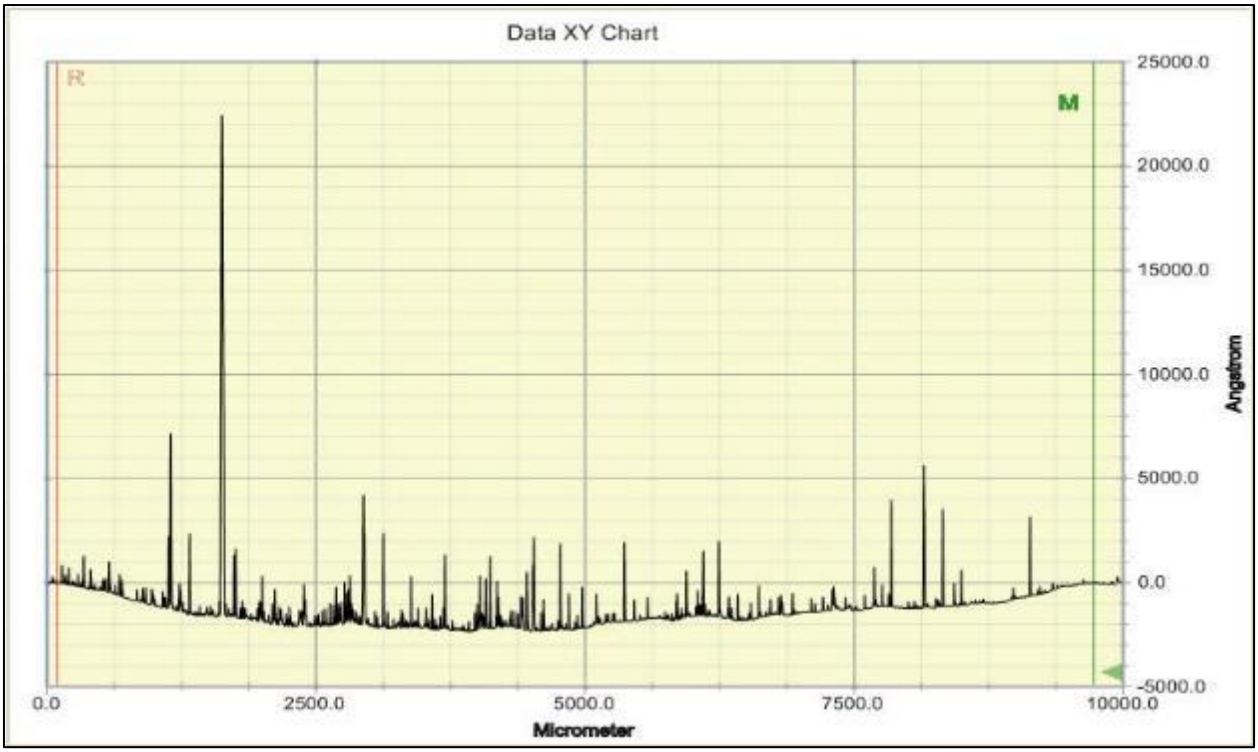


(b)

Figure S6 Two different Surface Studied Samples (a) S-4 (b) S-4(2).



(a)



(b)

Figure S7 Two different Surface Studied Samples (a) S-5(2) (b) S-5(4).