

# Microwave-Assisted Sol–Gel Preparation of the Nanostructured Magnetic System for Solid-Phase Synthesis

Daniela Istrati <sup>1</sup>, Alina Moroşan <sup>1</sup>, Raluca Stan <sup>1,\*</sup>, Bogdan Ştefan Vasile <sup>2</sup>, Gabriel Vasilievici <sup>3</sup>, Ovidiu Oprea <sup>4</sup>, Georgiana Dolete <sup>2</sup>, Bogdan Purcăreanu <sup>5</sup> and Dan Eduard Mihaiescu <sup>1,\*</sup>

<sup>1</sup> Department of Organic Chemistry “Costin Nenitescu”, Faculty of Applied Chemistry and Materials Science, Politehnica University of Bucharest, Bucharest 011061, Romania; daniela.istrati@upb.ro (D.I.); alina.morosan@upb.ro (A.M.)

<sup>2</sup> Department of Science and Engineering of Oxide Materials and Nanomaterials, Faculty of Applied Chemistry and Materials Science, Politehnica University of Bucharest, Bucharest 011061, Romania; vasile\_bogdan\_stefan@yahoo.com (B.Ş.V.); dolete.georgiana@gmail.com (G.D.)

<sup>3</sup> INCDCP-ICECHIM, 202 Spl. Independentei, Bucharest 011061, Romania; gabi.vasilievici@gmail.com

<sup>4</sup> Department of Inorganic Chemistry, Physical Chemistry and Electrochemistry, Faculty of Applied Chemistry and Materials Science, Politehnica University of Bucharest, Bucharest 011061, Romania; ovidiu73@yahoo.com

<sup>5</sup> S.C. BIOTEHNOS S.A., Gorunului Rue, No. 3–5, Ilfov 075100, Romania; bogdanpb89@gmail.com

\* Correspondence: raluca.stan@upb.ro (R.S.) and dan.mihaiescu@upb.ro (D.E.M.); Tel.: +40-7400-95101 (R.S.); +40-7290-35718 (D.E.M.)

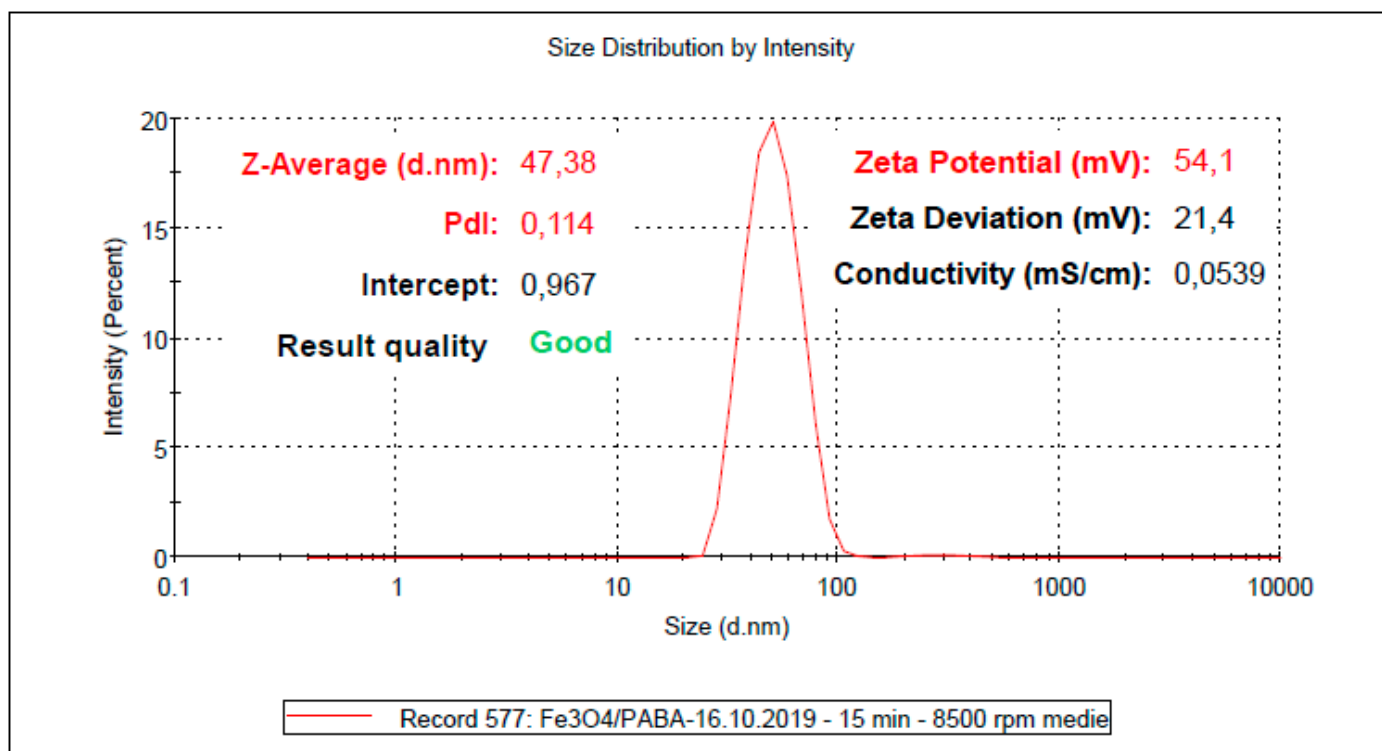
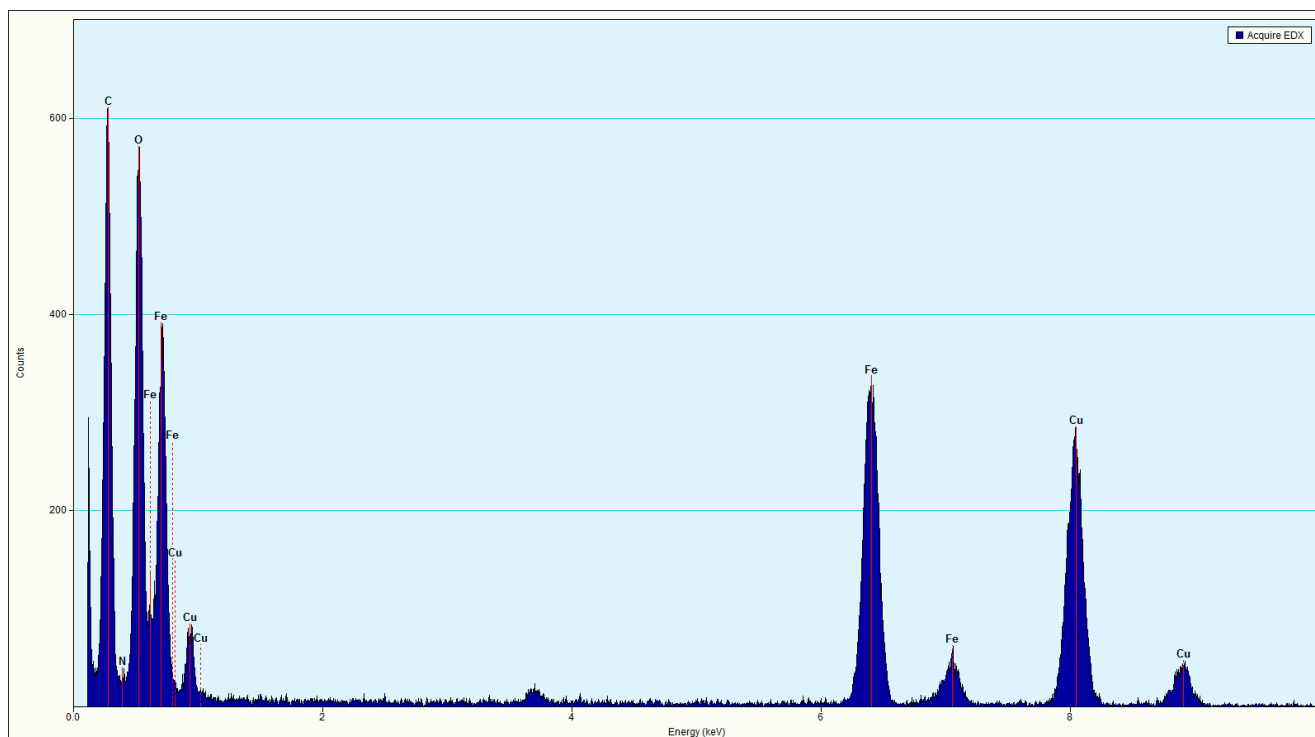
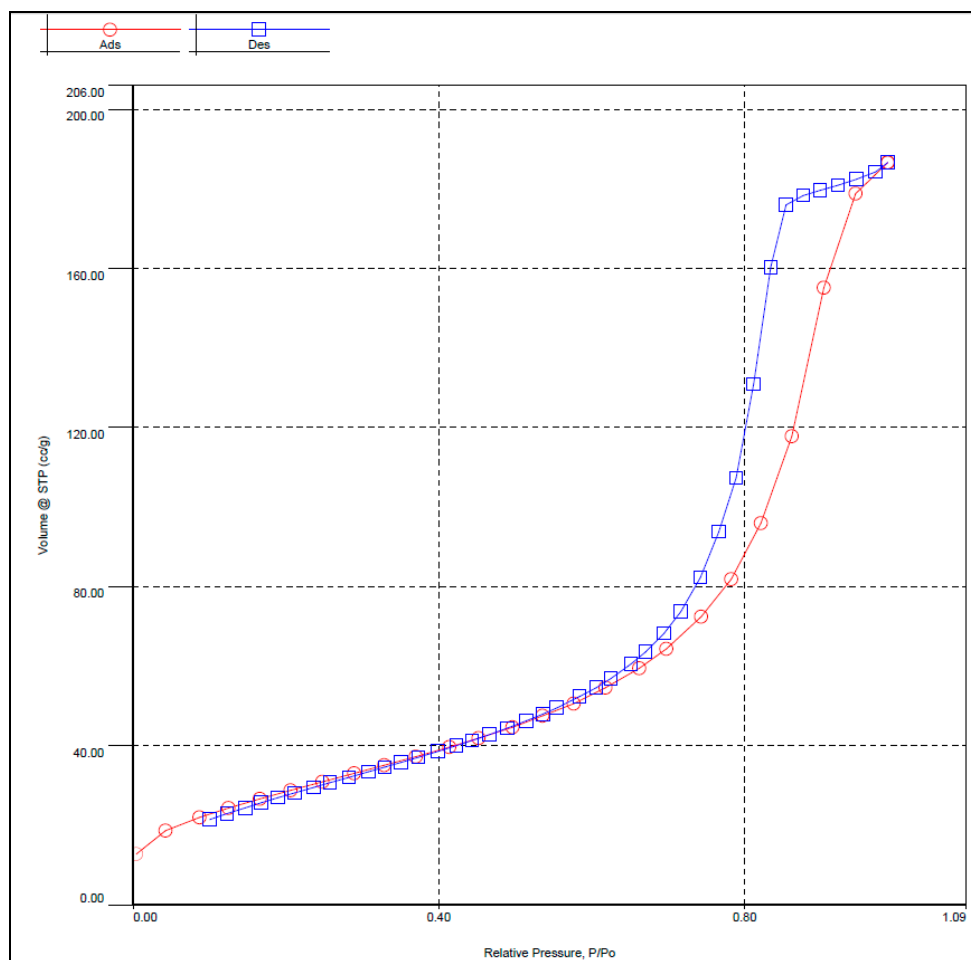


Figure S1. DLS analysis of magnetite nanoparticles with PABA shell.



**Figure S2.** The EDX spectrum for Fe<sub>3</sub>O<sub>4</sub>-PABA.



**Figure S3.** The N<sub>2</sub> adsorption/desorption isotherm for Fe<sub>3</sub>O<sub>4</sub>-PABA.

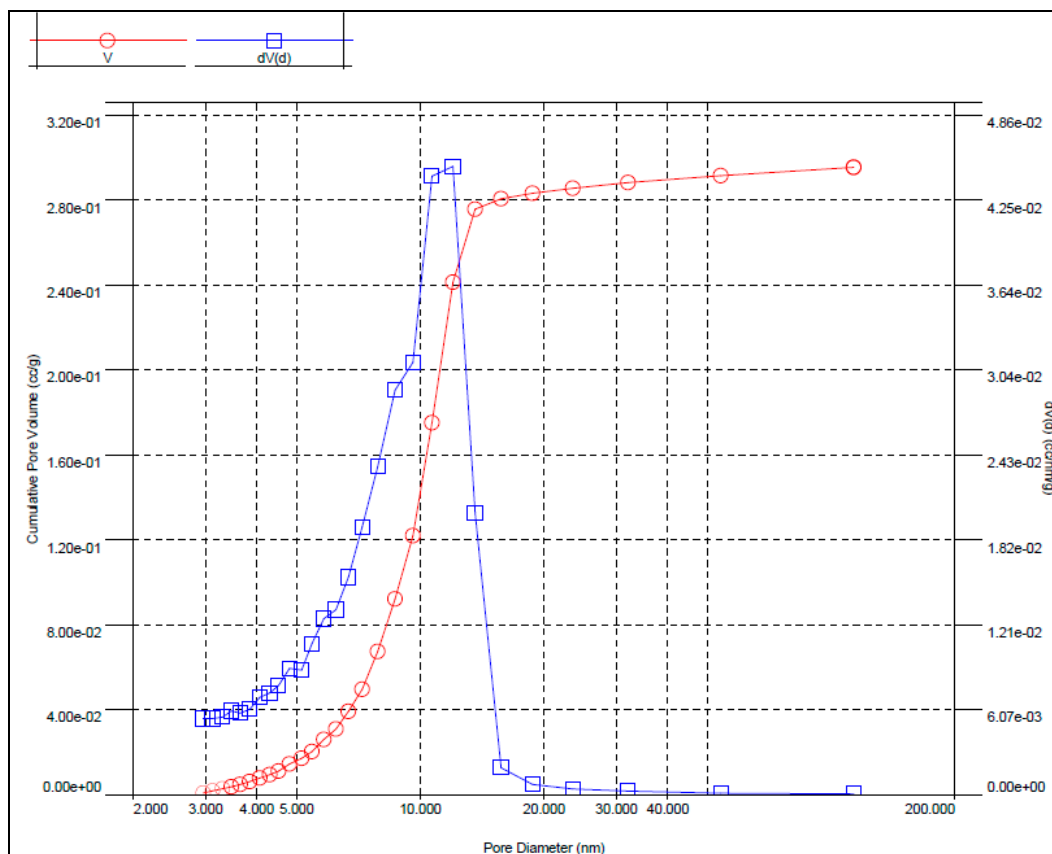


Figure S4. Pore size distributions for Fe<sub>3</sub>O<sub>4</sub>-PABA.

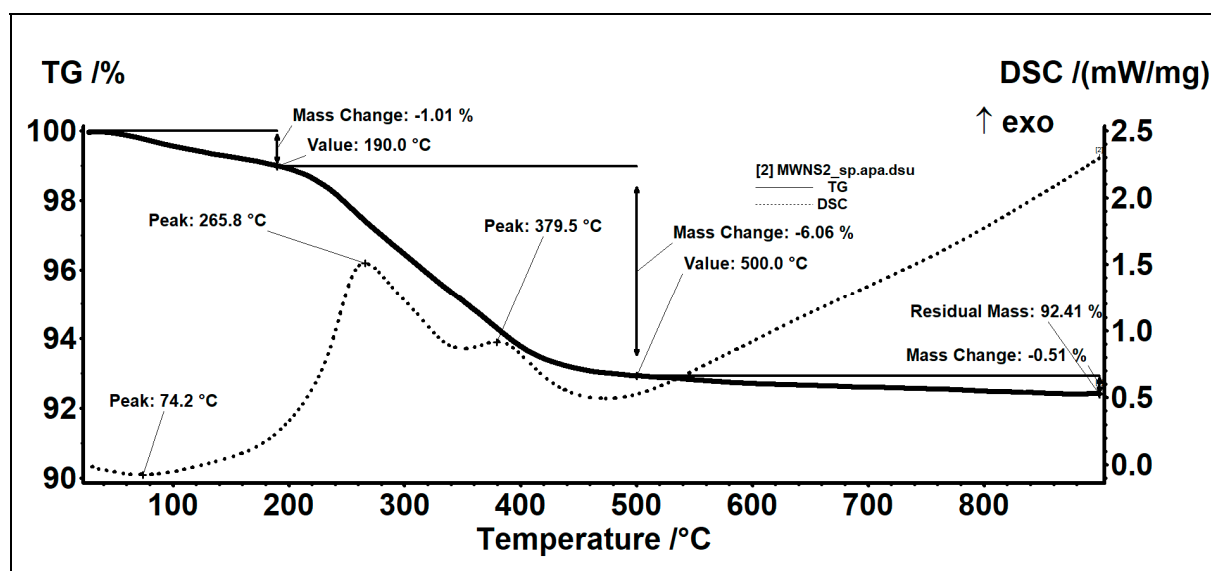


Figure S5. TGA analysis of the Fe<sub>3</sub>O<sub>4</sub>-PABA-SiO<sub>2</sub> nanoparticles after the water washing step.

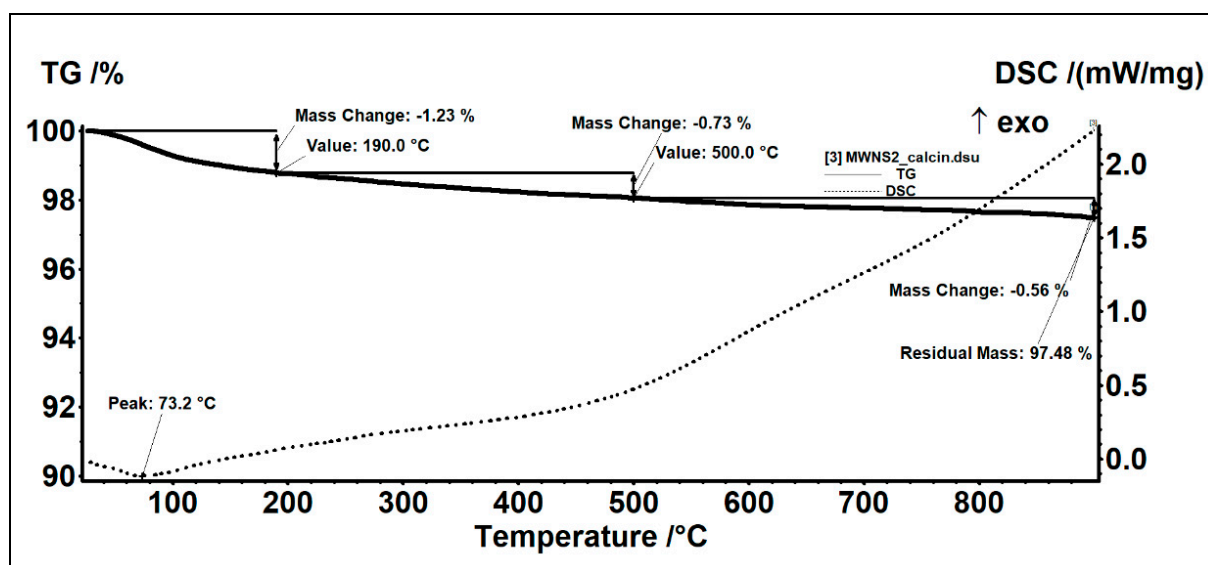


Figure S6. TGA analysis of the MW-NPS-1 nanoparticles.

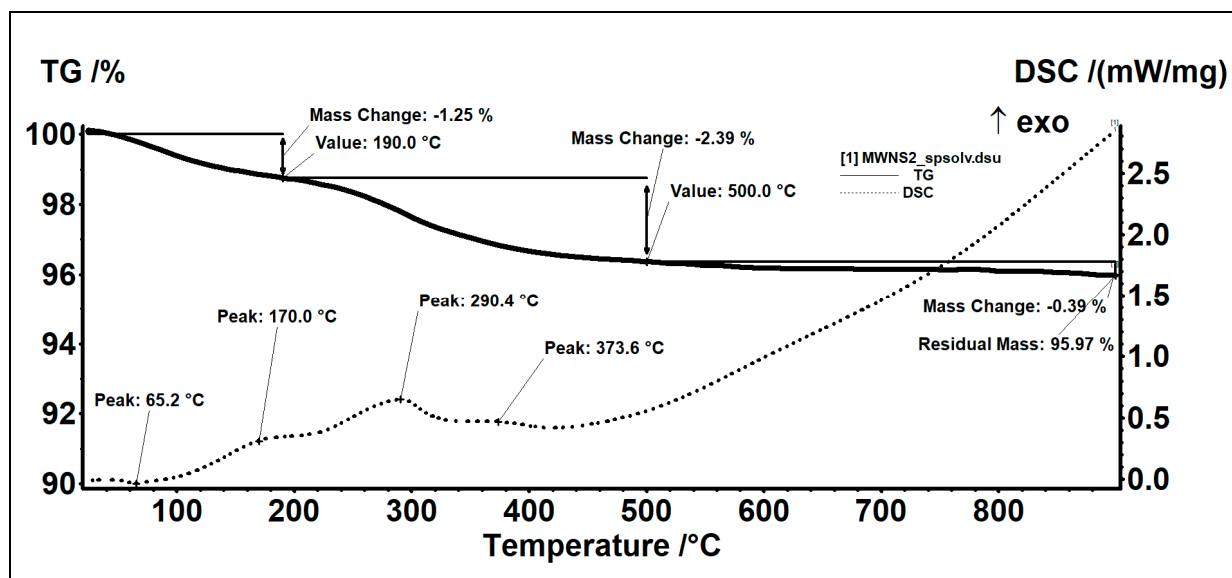
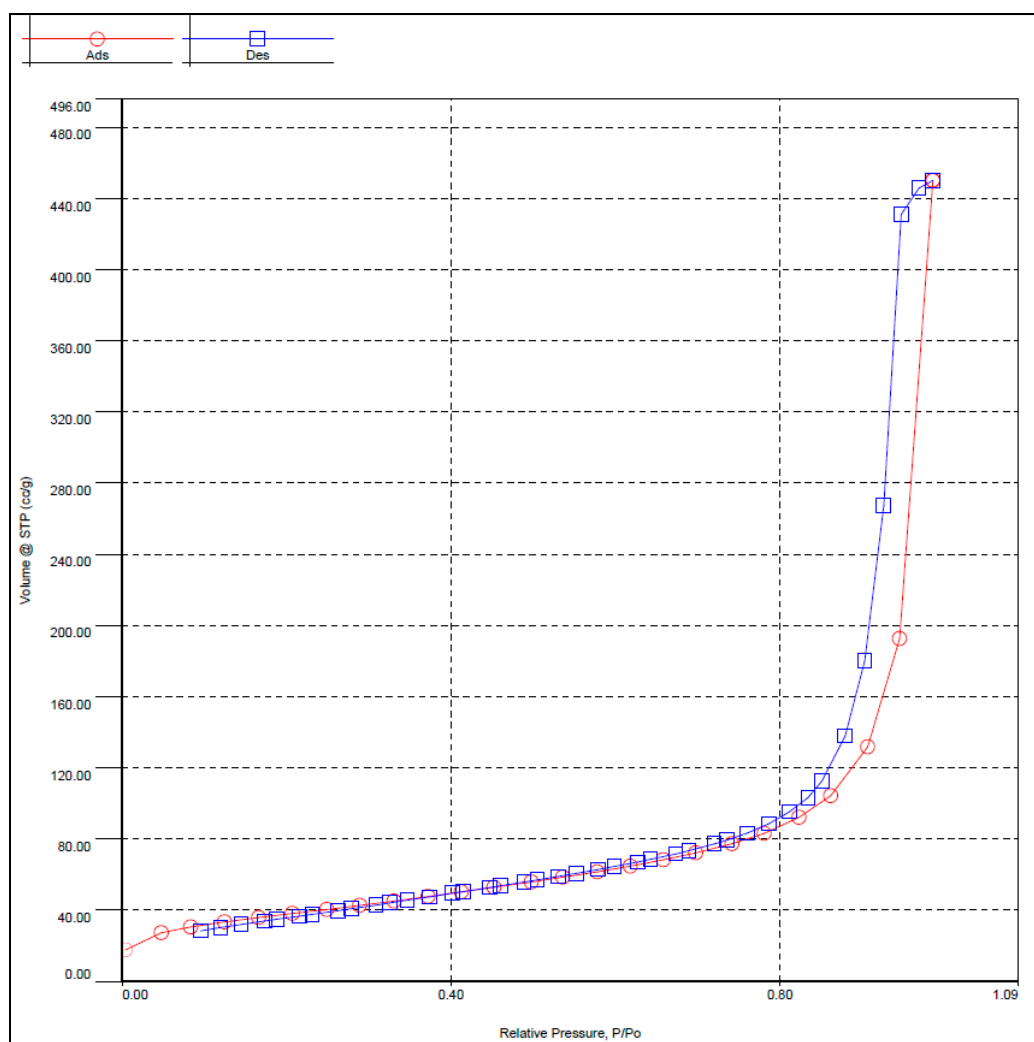
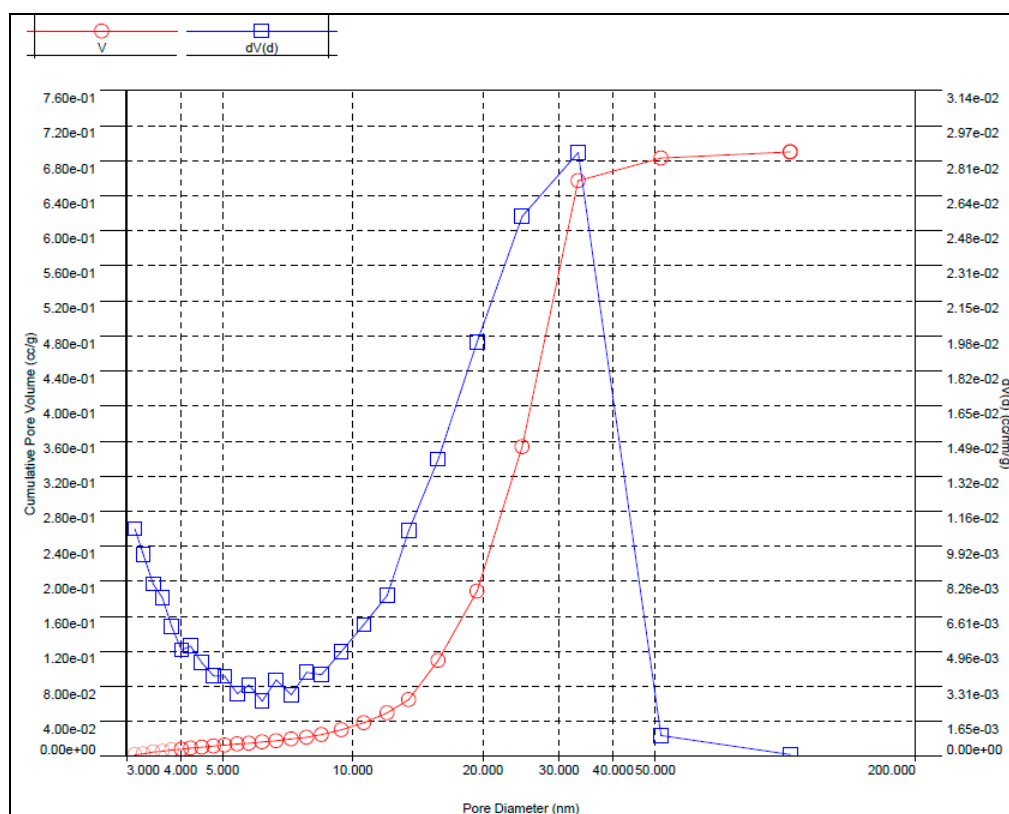


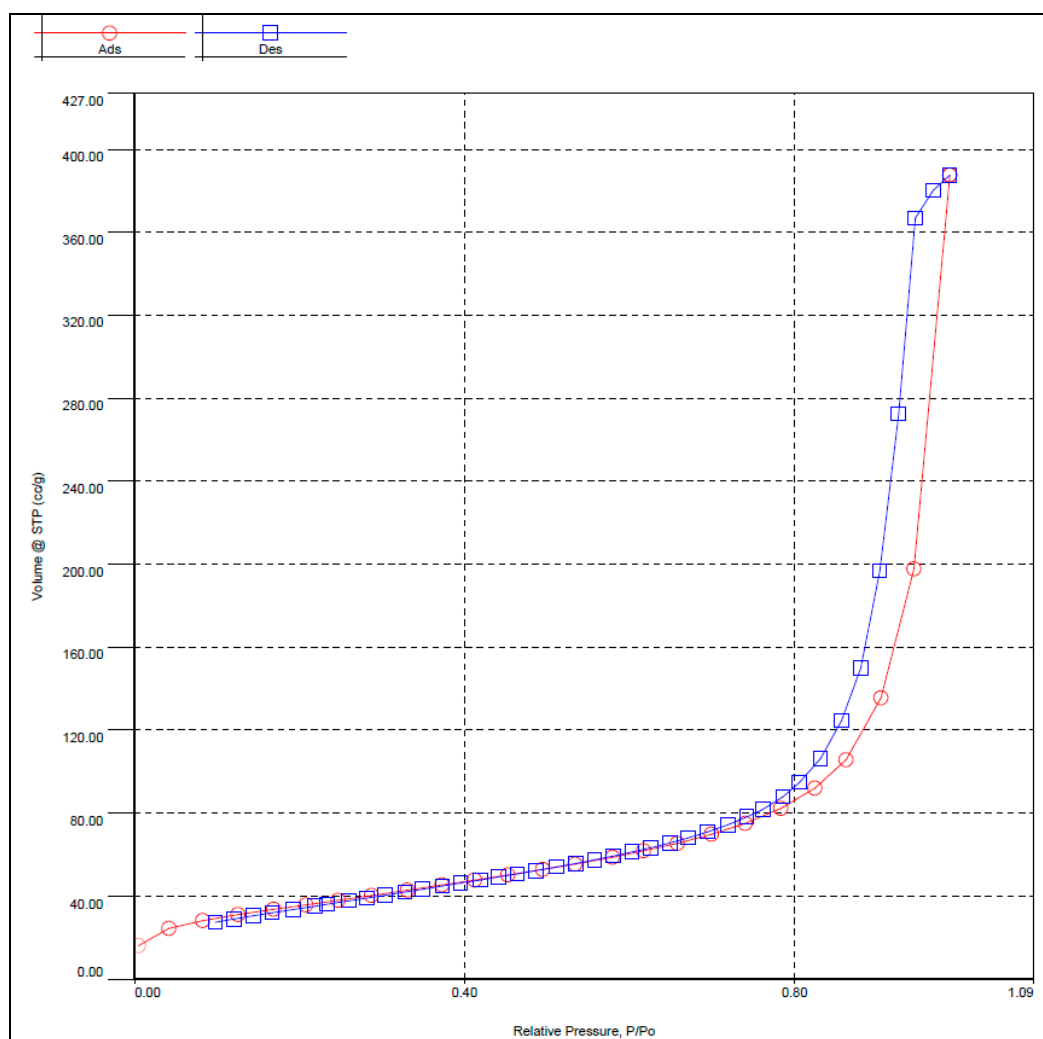
Figure S7. TGA analysis of the MW-NPS-2 nanoparticles.



**Figure S8.** The N<sub>2</sub> adsorption/desorption isotherm for MW-NPS-1.



**Figure S9.** Pore size distributions for MW-NPS-1.



**Figure S10.** The N<sub>2</sub> adsorption/desorption isotherm for MW-NPS-2.

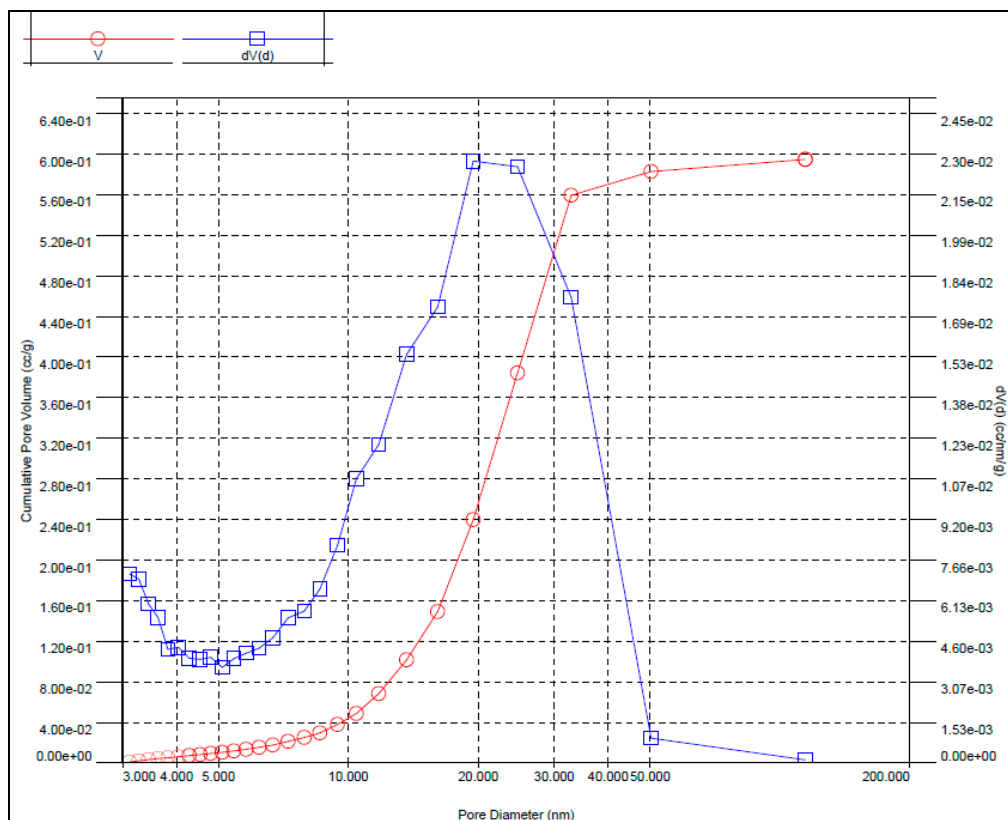


Figure S11. Pore size distributions for MW-NPS-2.

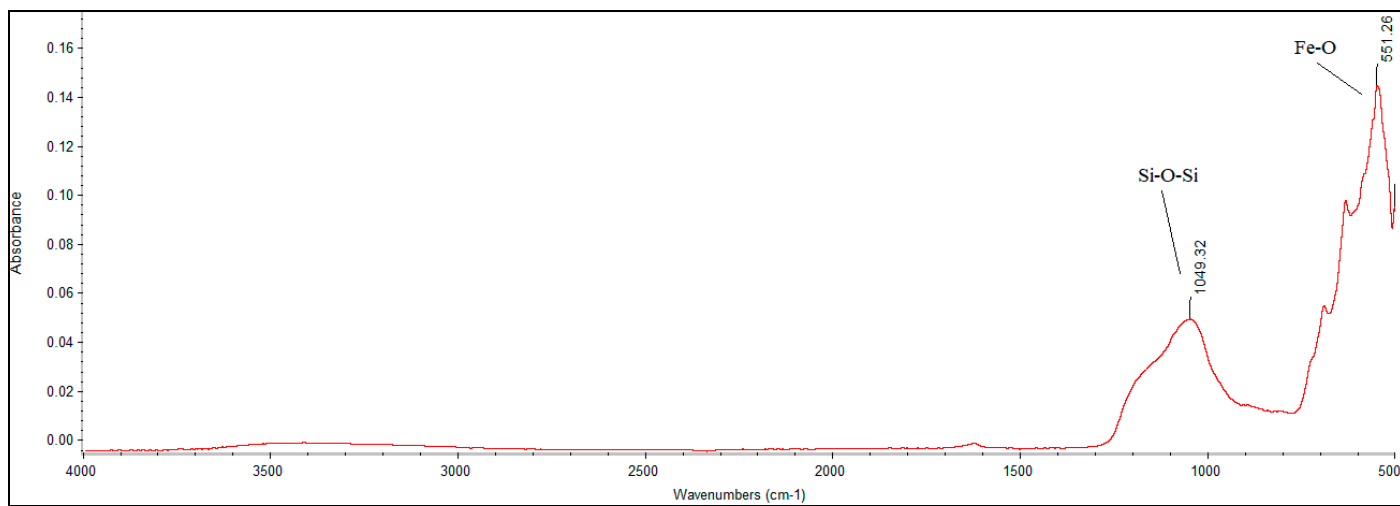


Figure S12. The FT-IR spectrum of MW-NPS-1.



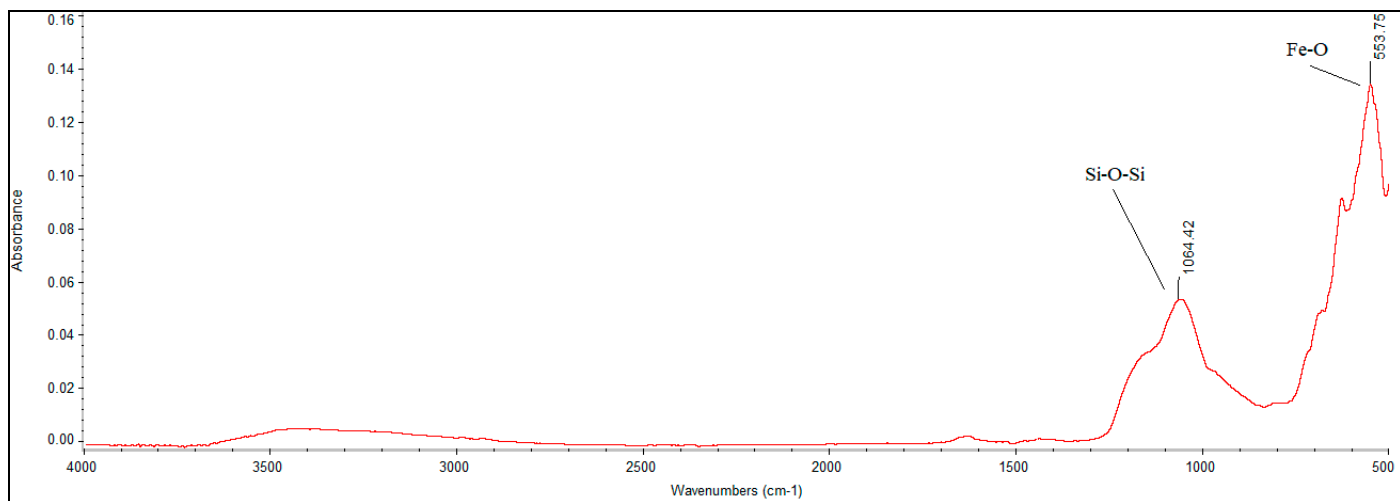


Figure S13. The FT-IR spectrum of MW-NPS-2.

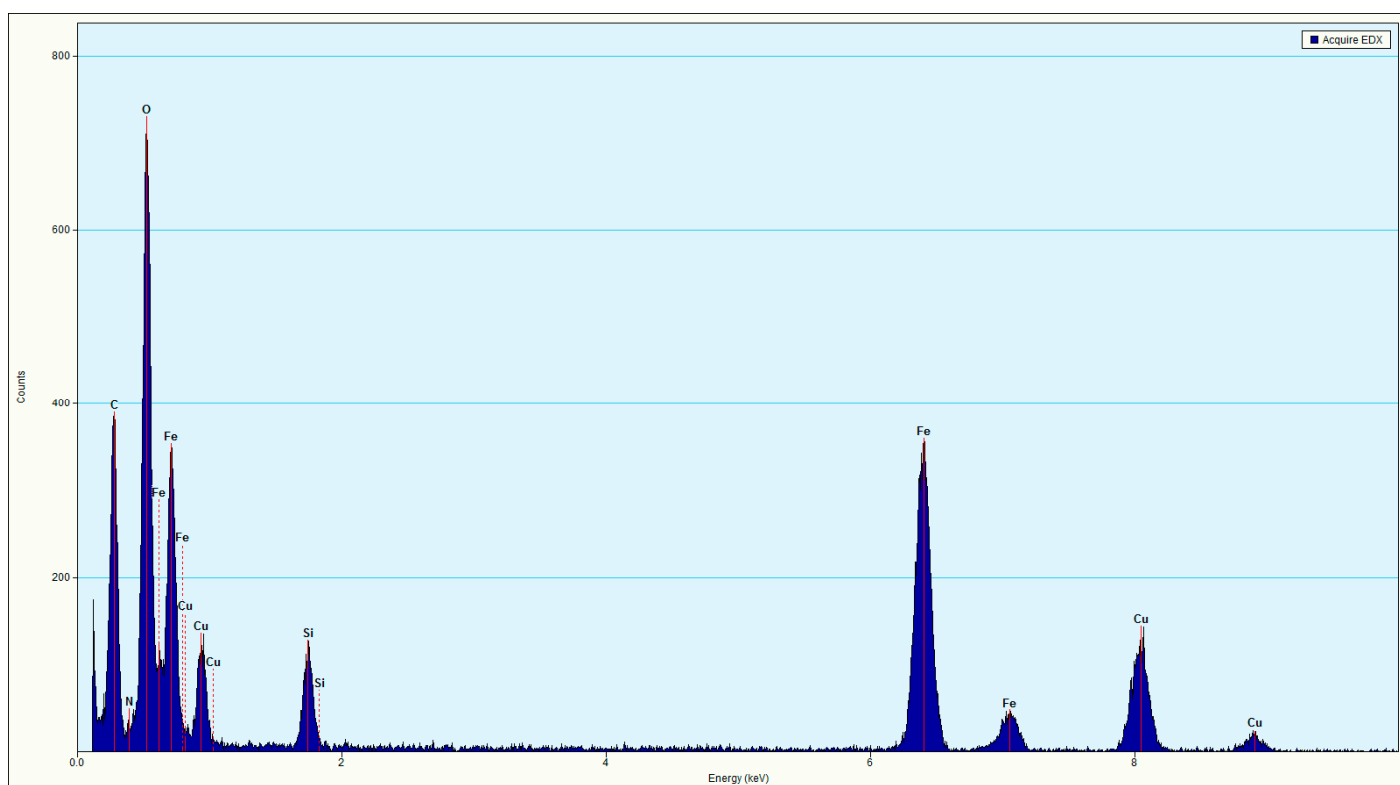


Figure S14. The EDX spectrum for MW-NPS-1.

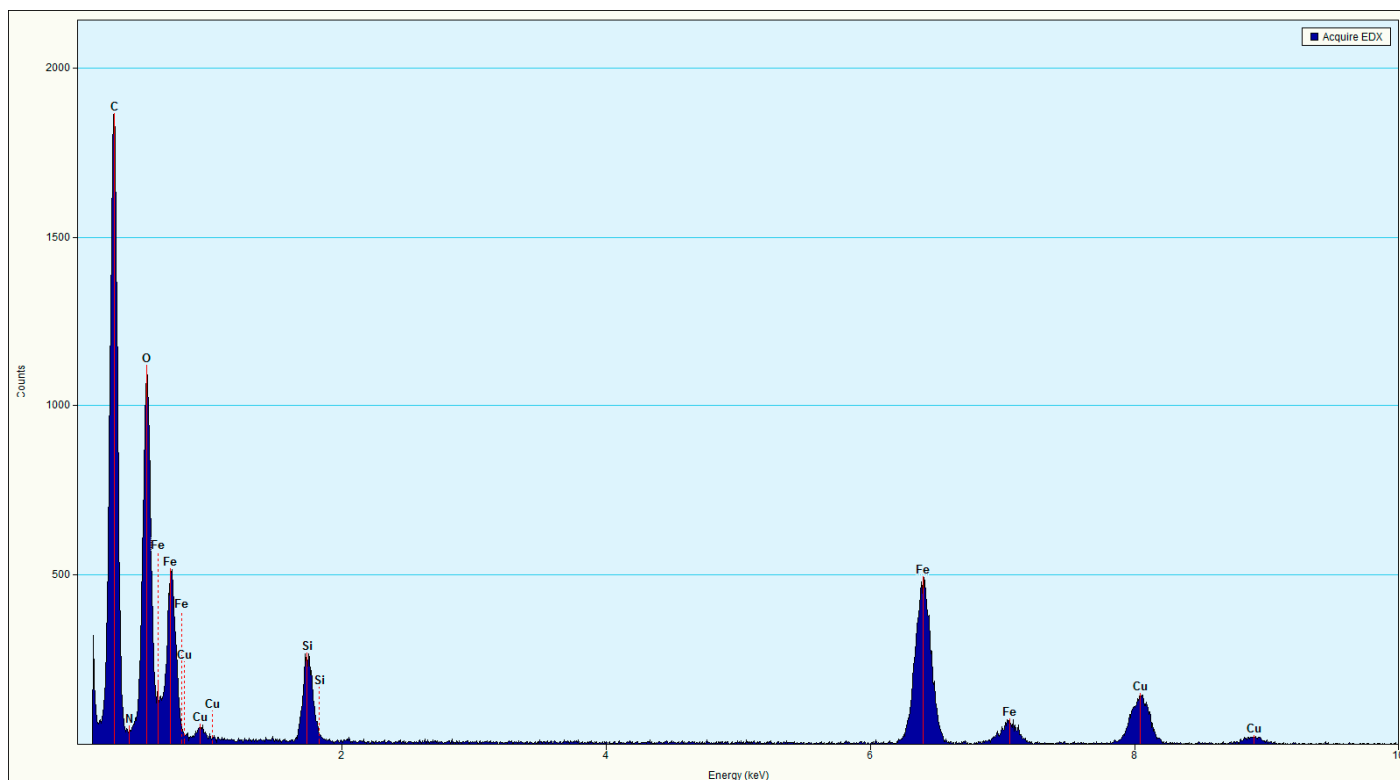


Figure S15. The EDX spectrum for MW-NPS-2.

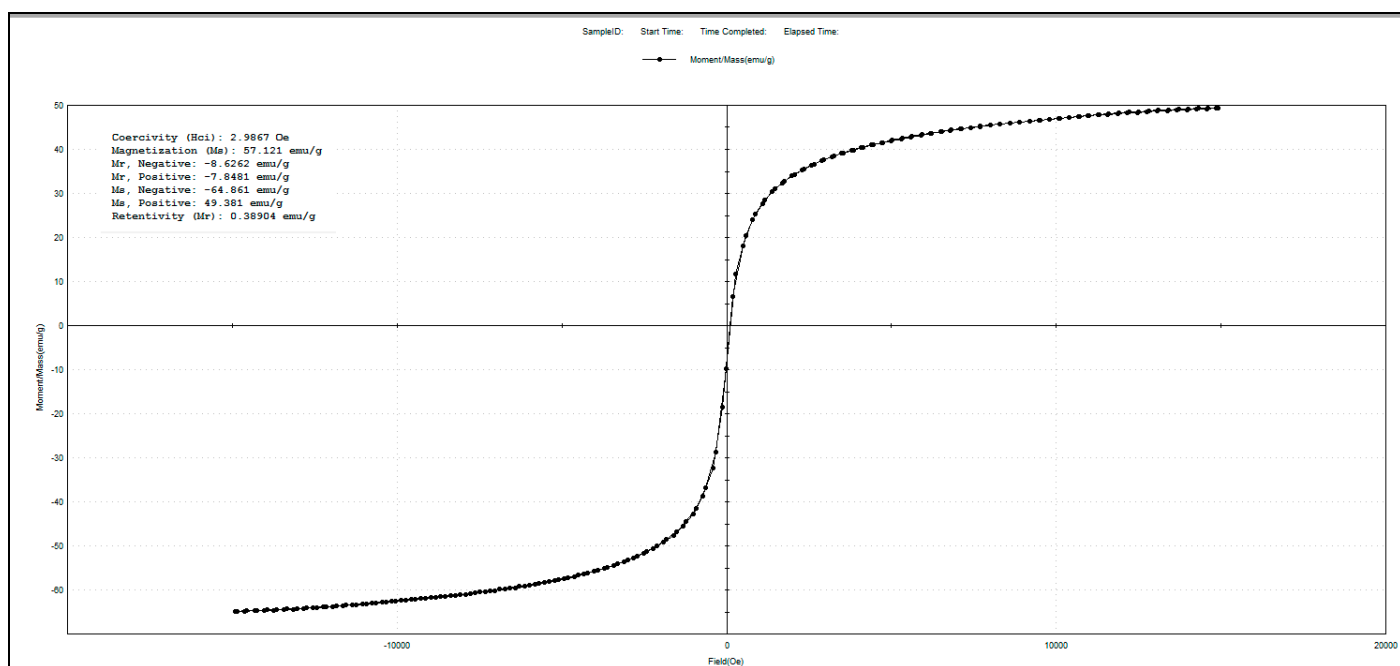
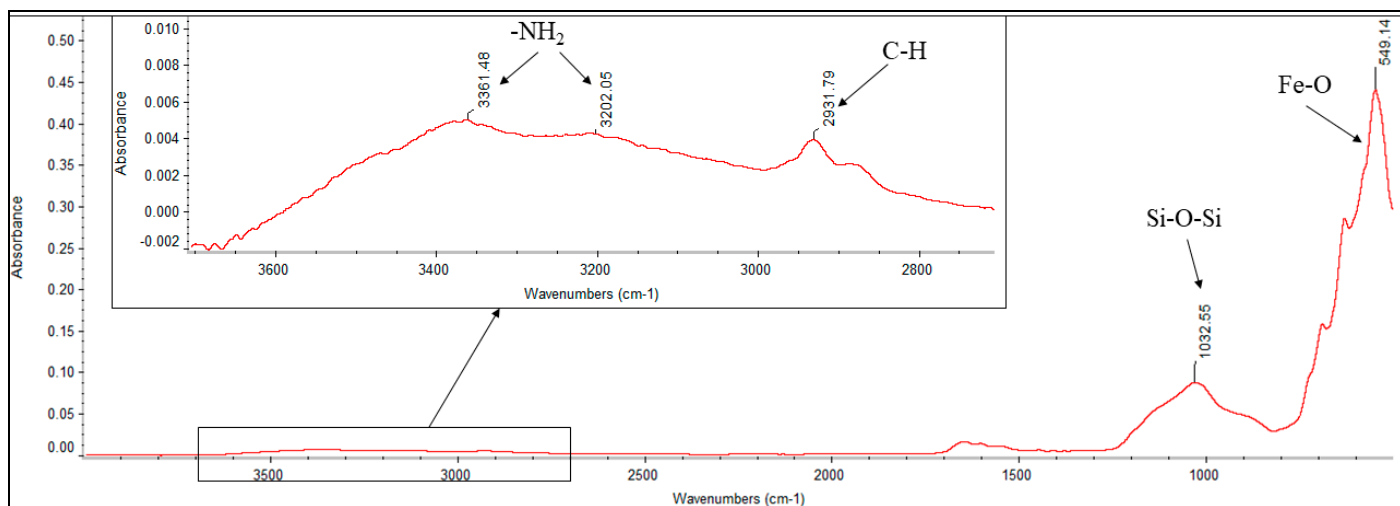
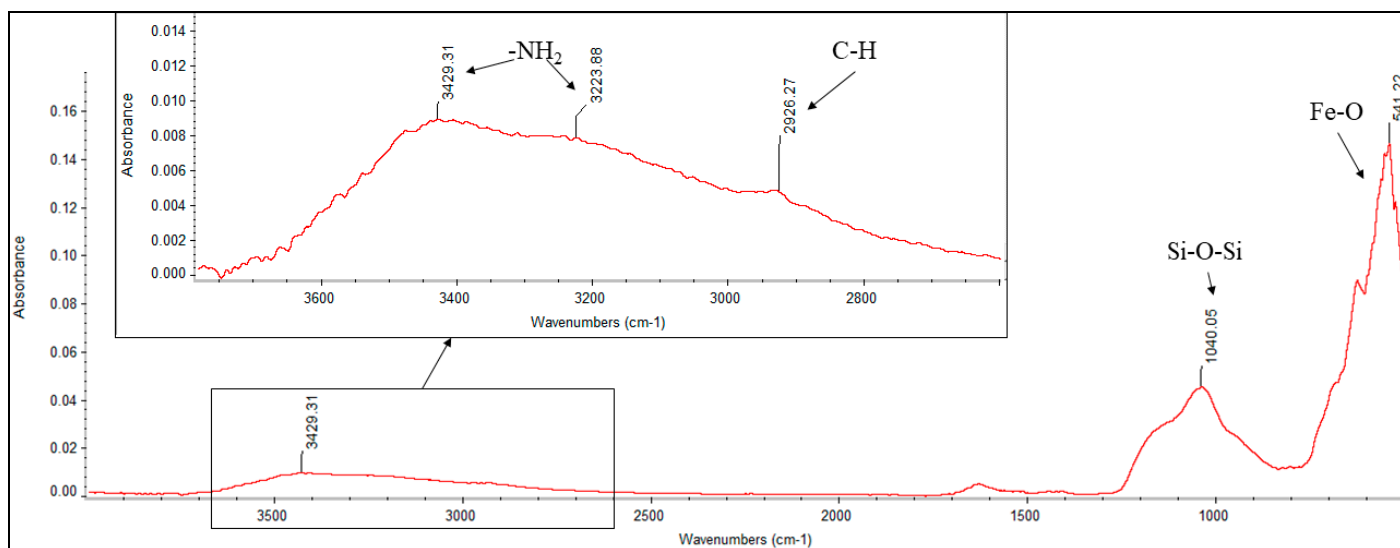


Figure S16. Magnetization curve of MW-NPS-2 nanoparticles.



**Figure S17.** The FT-IR spectrum of MW-NPS-1-APS.



**Figure S18.** The FT-IR spectrum of MW-NPS-2-APS.

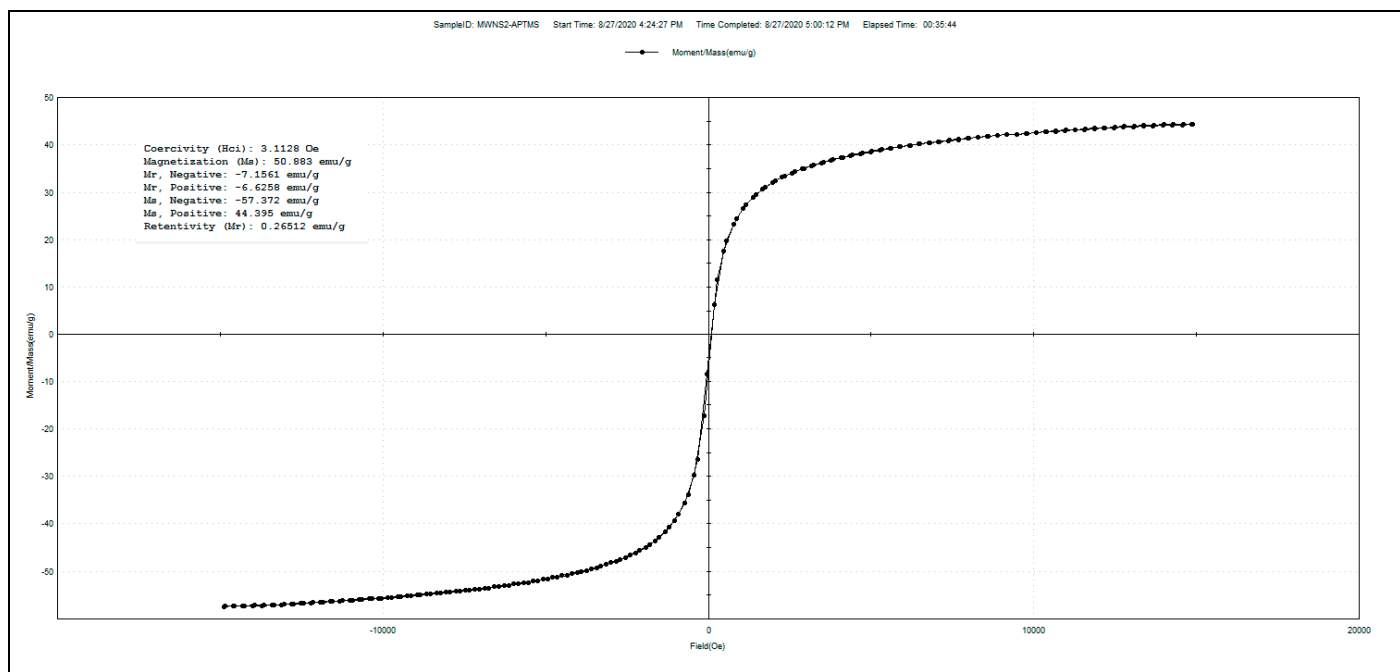


Figure S19. Magnetization curve of MW-NPS-2-APS.

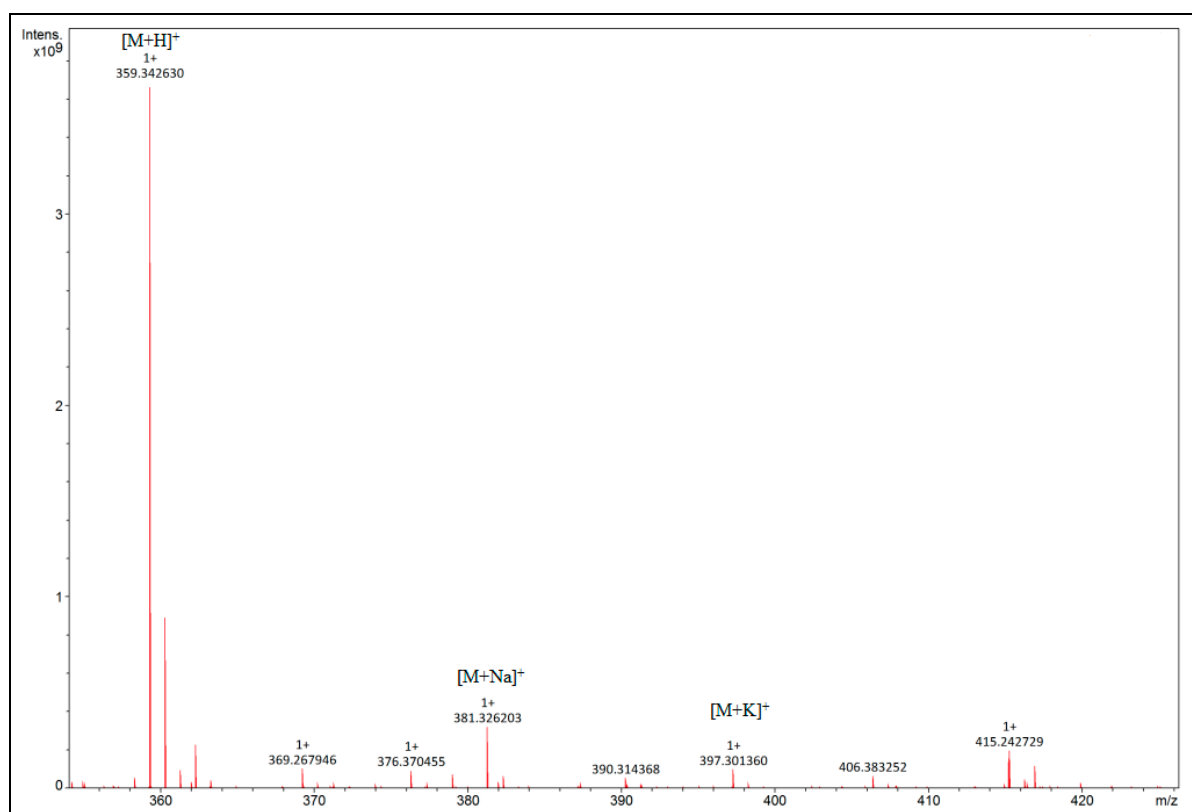
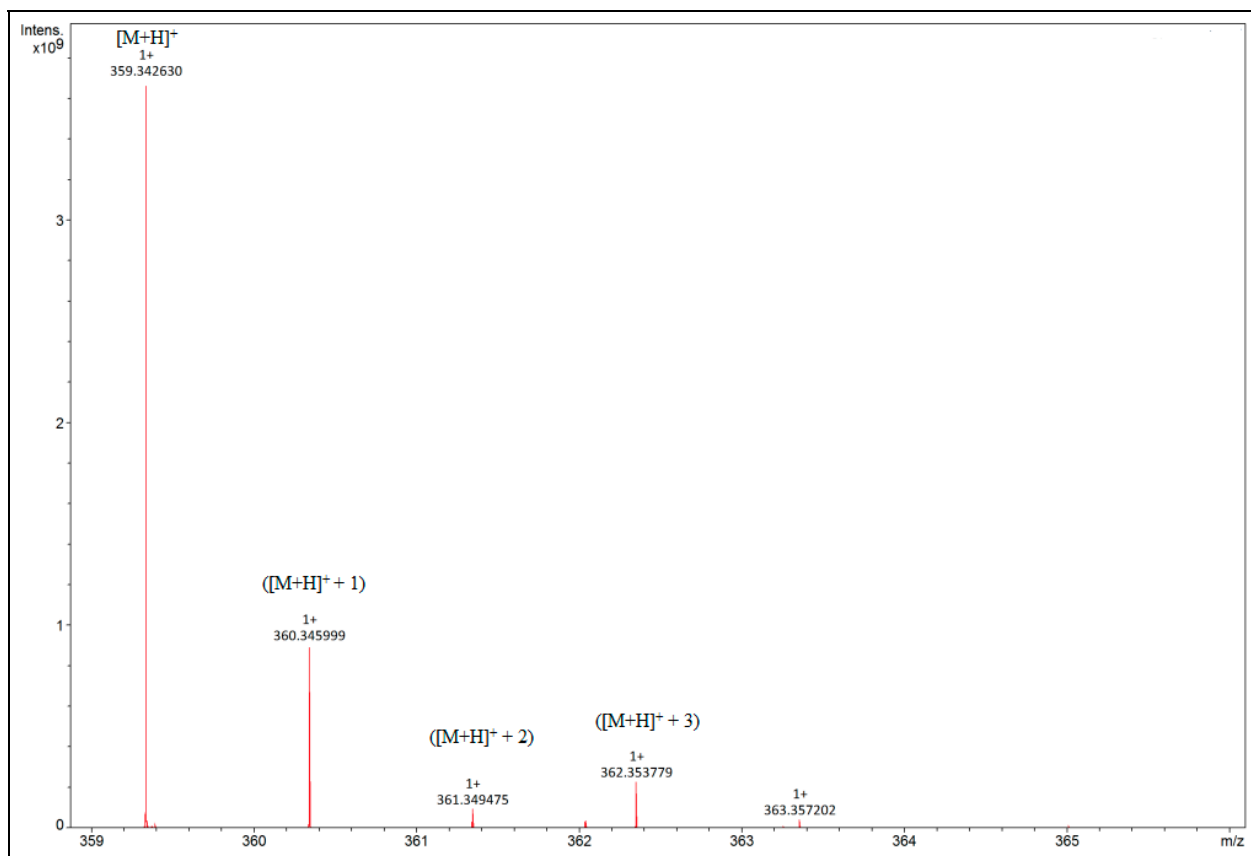
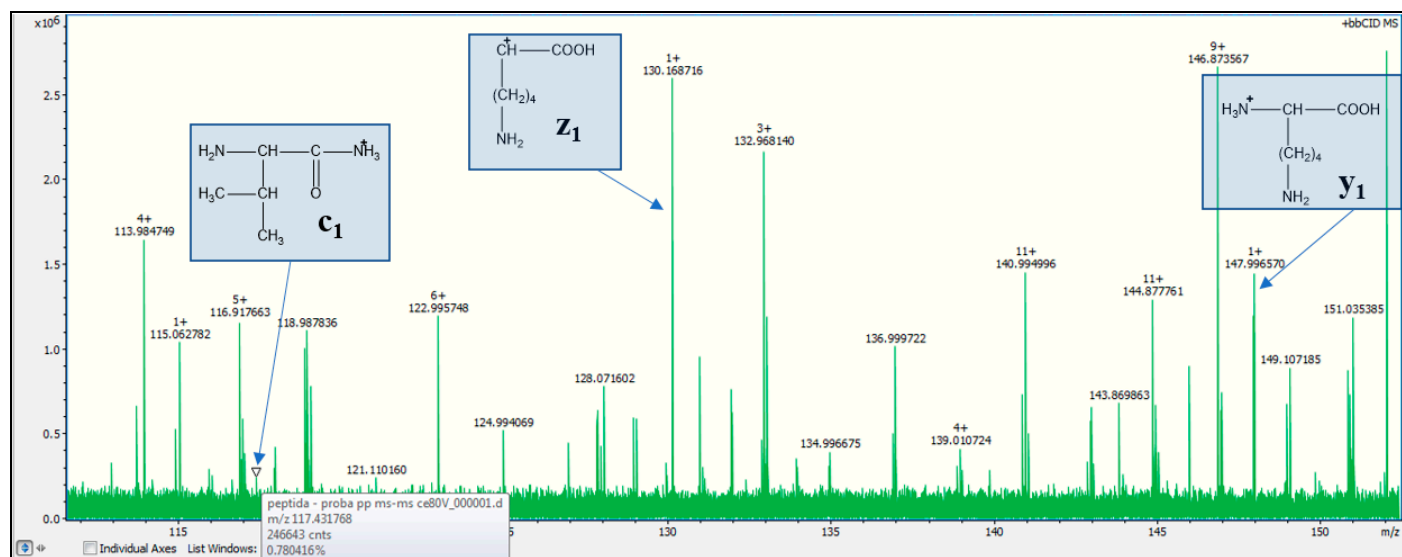


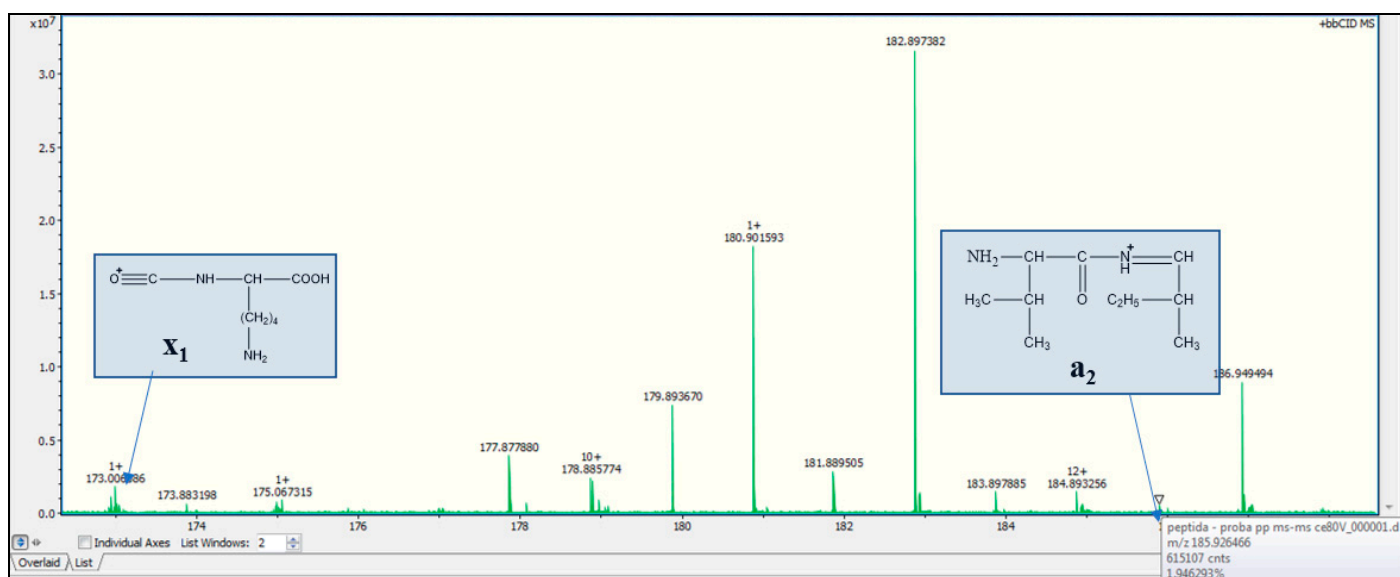
Figure S20. HR-MS analysis of peptide Val-Ile-Lys after cleavage from the NW-NPS-2-APS-HMBA nanostructured system.



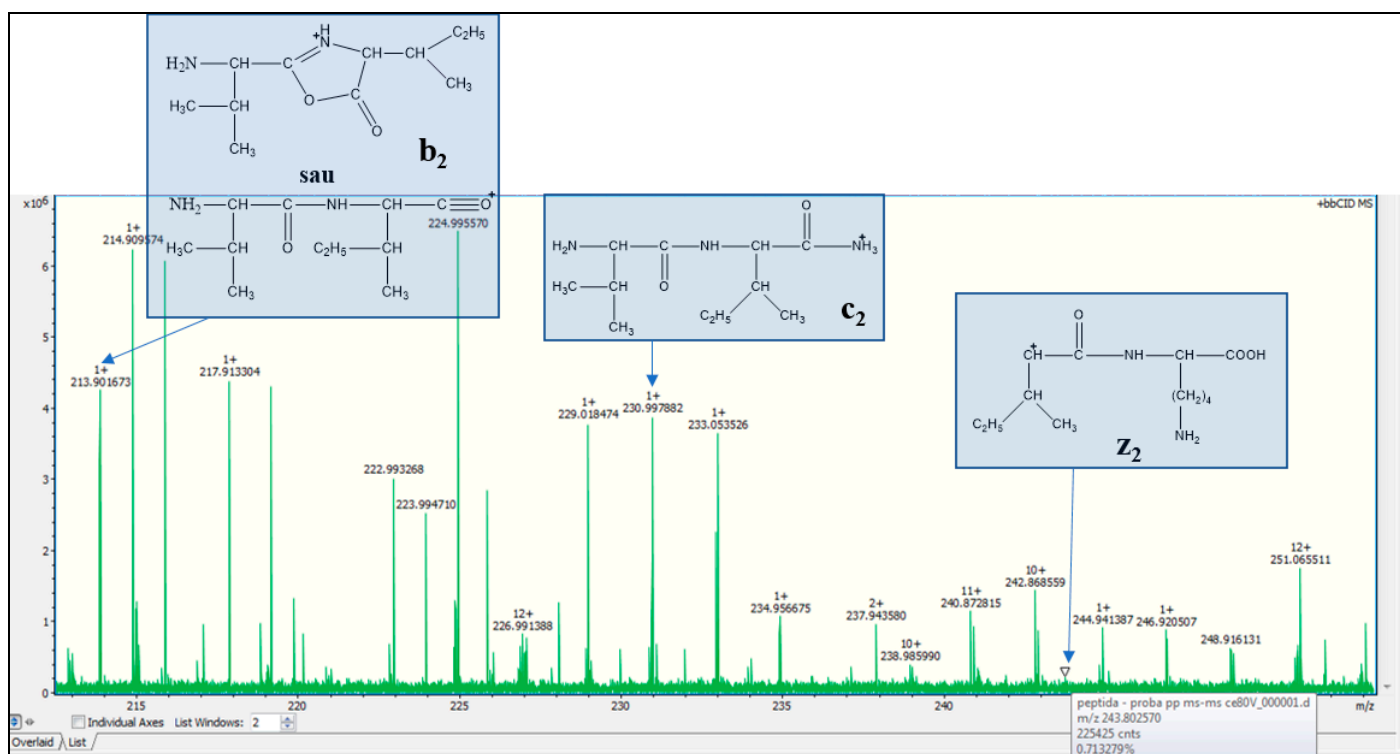
**Figure S21.** HR-MS analysis of peptide Val-Ile-Lys. Highlighting of ([M+H]<sup>+</sup> + 1), ([M+H]<sup>+</sup> + 2), ([M+H]<sup>+</sup> + 3) type isotopic peaks derived from the protonated molecular ion [M+H]<sup>+</sup>.



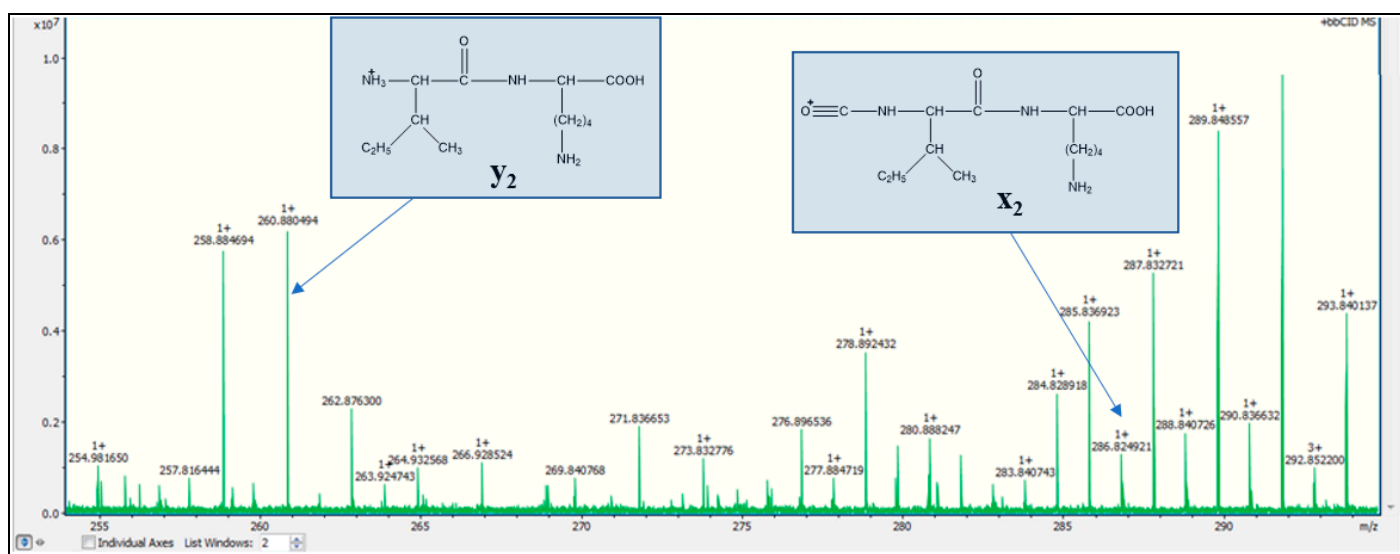
**Figure S22.** Identification in the MS-MS spectrum (from 359.34 monoisotopic peak precursor) of c<sub>1</sub>, y<sub>1</sub>, z<sub>1</sub> type fragments with m/z 117.43, 130.16, respectively 147.99.



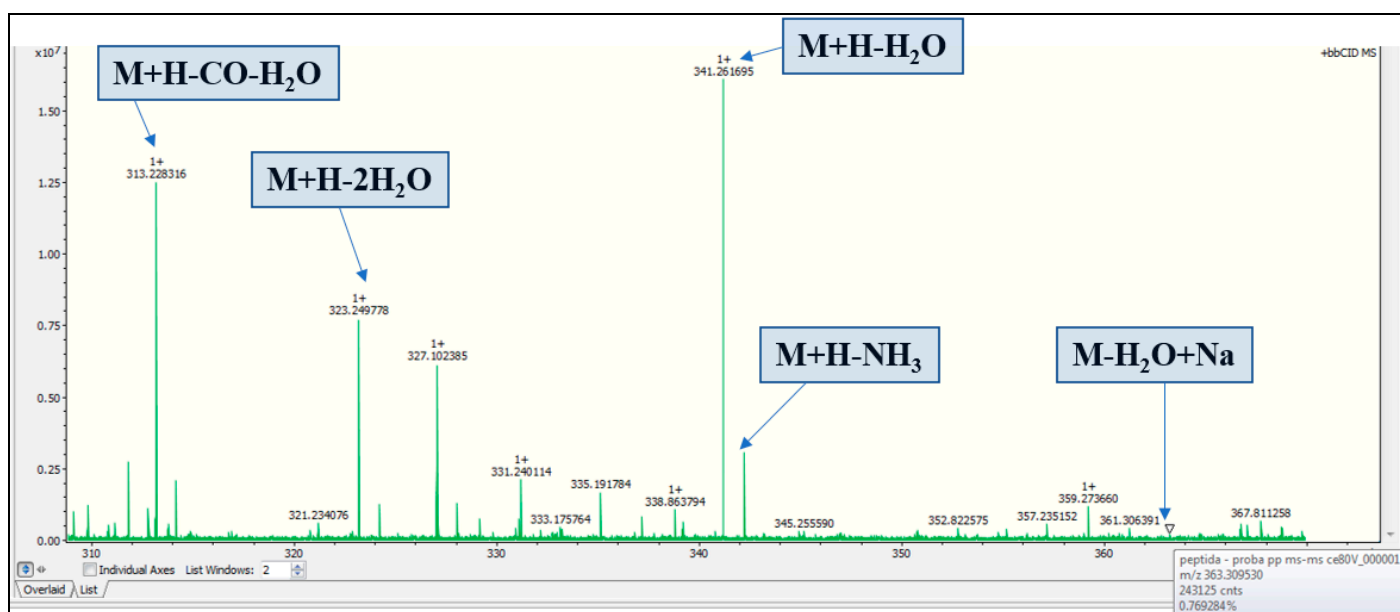
**Figure S23.** Identification in the MS-MS spectrum (from 359.34 monoisotopic peak precursor) of  $x_1$  and  $a_2$  type fragments with  $m/z$  173.00, respectively 185.92.



**Figure S24.** Identification in the MS-MS spectrum (from 359.34 monoisotopic peak precursor) of  $b_2$ ,  $c_2$  and  $z_2$  type fragments with  $m/z$  213.90, 230.99, respectively 243.80.



**Figure S25.** Identification in the MS-MS spectrum (from 359.34 monoisotopic peak precursor) of  $x_2$  and  $y_2$  type fragments with  $m/z$  286.82 and 260.88.



**Figure S26.** Identification in the MS-MS spectrum (from 359.34 monoisotopic peak precursor) of  $[M+H-H_2O]^+$ ,  $[M+H-CO-H_2O]^+$ ,  $[M+H-NH_3]^+$ ,  $[M-H_2O+Na]^+$ ,  $[M+H-2H_2O]^+$  type fragments with  $m/z$  341.26, 313.22, 342.26, 363.30, respectively 323.24.