Hazerfen

Kimya Malzeme ve Enerji Teknolojileri Sanayi Ticaret A.Ş.

TECHNICAL LEAFLET

GRAPHENE OXIDE

X-Ray Diffraction Pattern of Graphene Oxide (GO)

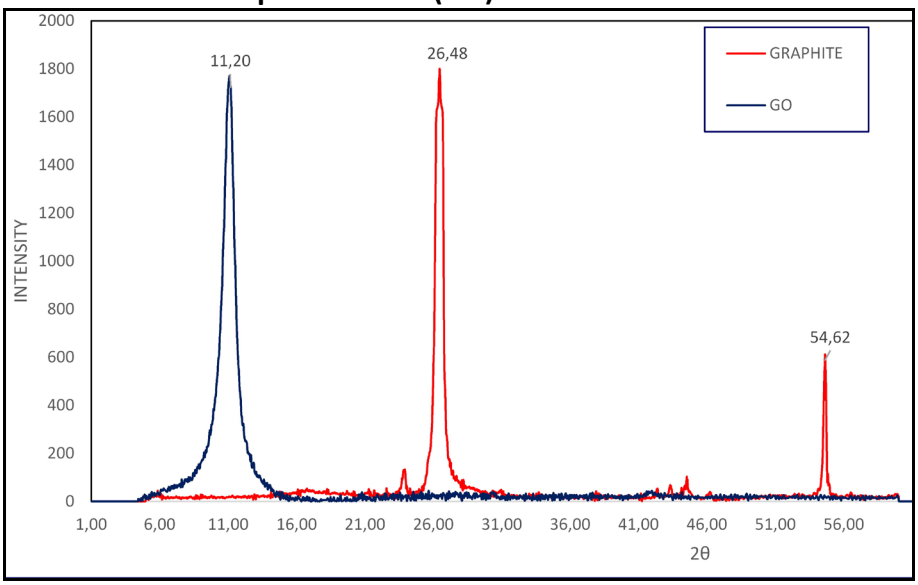


Fig. 1 X-ray diffraction patterns of graphite and synthesized GO

Graphite flakes exhibits a strong and sharp peak at 26.48° in Fig.1 indicating a higher ordered structure, that corresponds to a basal spacing d_{002} = 0.334nm. The pattern of graphene oxide, on the other hand, exhibits a 001 reflection at 11.20 corresponding to a basal spacing of d_{001} =0.782nm. This value is higher than interlayer spacing of graphite flakes (d-spacing= 0.334nm, 2Θ =26.4°), due to the presence of oxygenated functional groups and intercalated water molecules. XRD patterns of GO is given in Fig. 1. GO shows characteristic GO peak between 2θ=9-11°.

EDX-EDS Analysis of Graphene Oxide (GO)

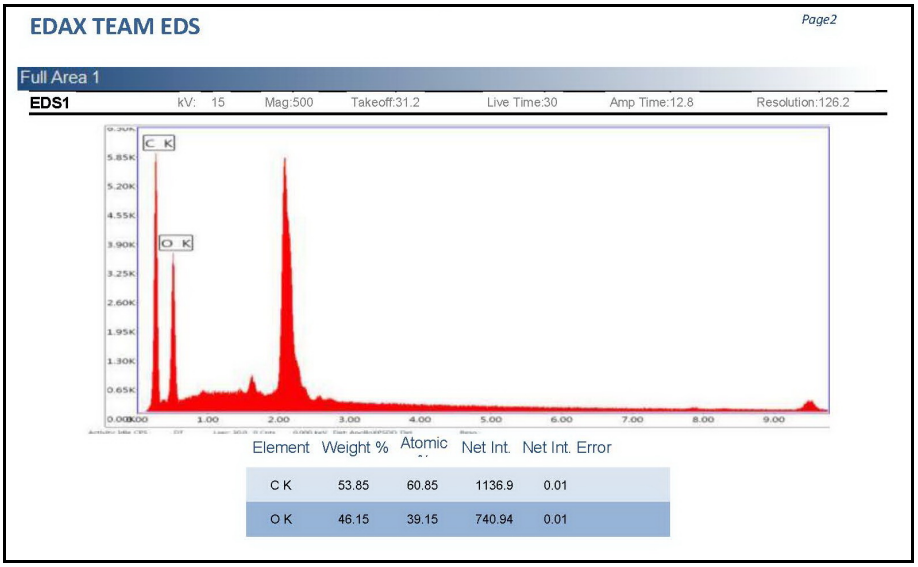


Fig. 2 EDS-EDX analysis of GO

The atomic C: O ratio of GO derived from EDX analysis is 1,55. The oxidation degree of GO was calculated by using C:O atomic ratio.

SEM Images of Graphite and Graphene Oxide (GO)

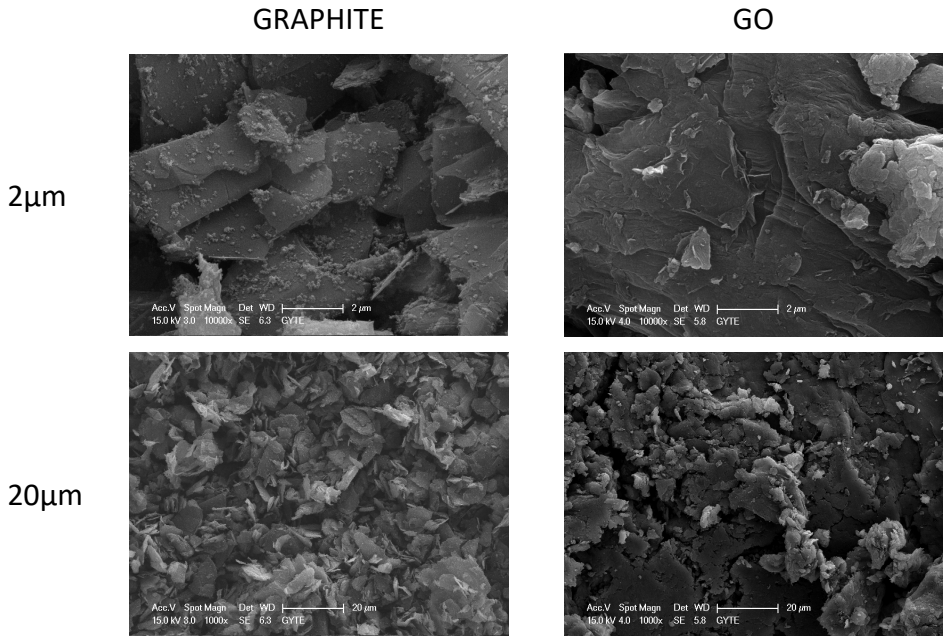


Fig. 3 SEM Images of Graphite and GO with 2 and 20 µm scale.

Morphological appearances of graphite and GO obtained from SEM images were given in Fig. 3. As seen from SEM images, the morphological appearance was altered.

FTIR Spectra of Graphite and Graphene Oxide (GO)

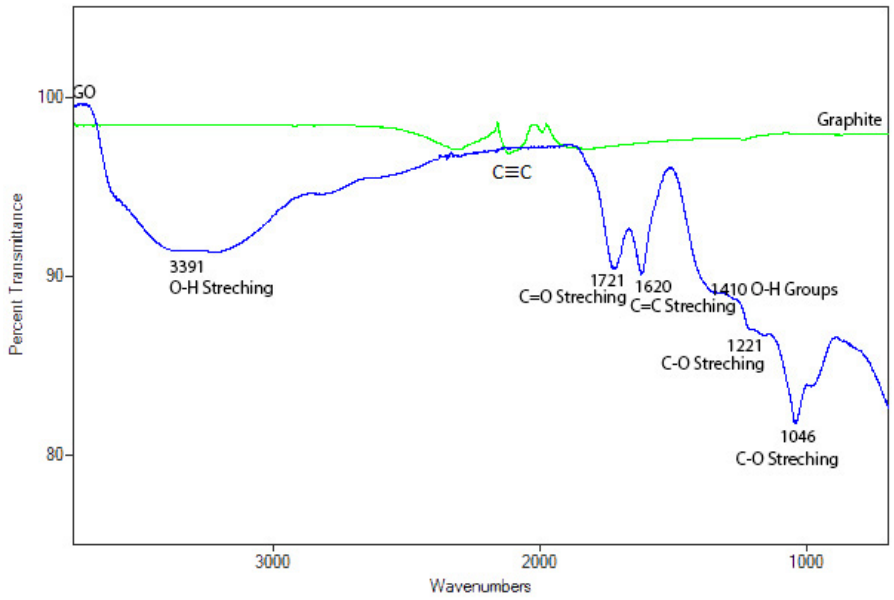


Fig. 4 FT-IR spectrum of GO (blue) and graphite (green)

It was assumed that various functional groups including oxygen were tied on GO flakes during oxidation of graphite. These functional structures are hydroxyl and epoxy groups on the basal plane, with a smaller amount of carboxy, carbonyl at the sheet edges.

As seen in Fig.4, The band observed at 1721 cm $^{-1}$ is assigned to the C=O stretching vibration. The vibration and deformation peaks of O–H groups at 3391 cm $^{-1}$ and 1410 cm $^{-1}$, the C–O (epoxy) stretching vibration peak at 1221 cm^{-1}, the C–O (alkoxy) stretching peak at 1046 cm$^{-1,}$ and Alkenyl C=C stretching peak at 1680 - 1620 cm $^{-1}$ were observed on FT-IR spectrum. The spectrum indicates binding of the oxygen containing groups on graphite to form GO.

TGA Analysis of Graphene Oxide (GO)

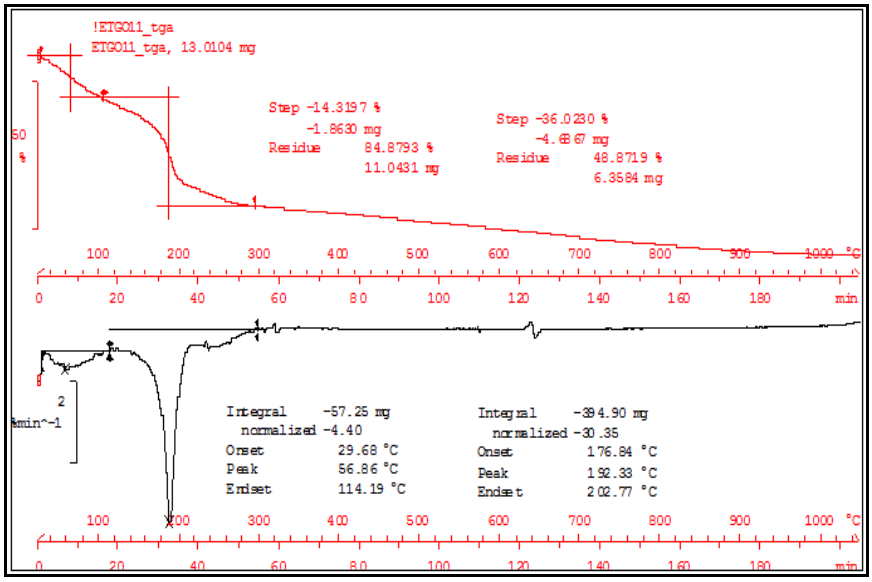


Fig. 5 TGA analysis of GO.

Although GO is thermally unstable and starts to lose mass upon heating even below 100 °C , the major mass loss occurs at 200 °C, presumably due to pyrolysis of the labile oxygen-containing functional groups, yielding CO,CO_2, and steam. Hence, the thermal decomposition of GO can be accompanied by a vigorous release of gas, resulting in a rapid thermal expansion of the material. This is evident by both a large volume expansion and a larger mass loss. (Fig. 5)

DXR Raman Spectra of Graphite and Graphene Oxide (GO)

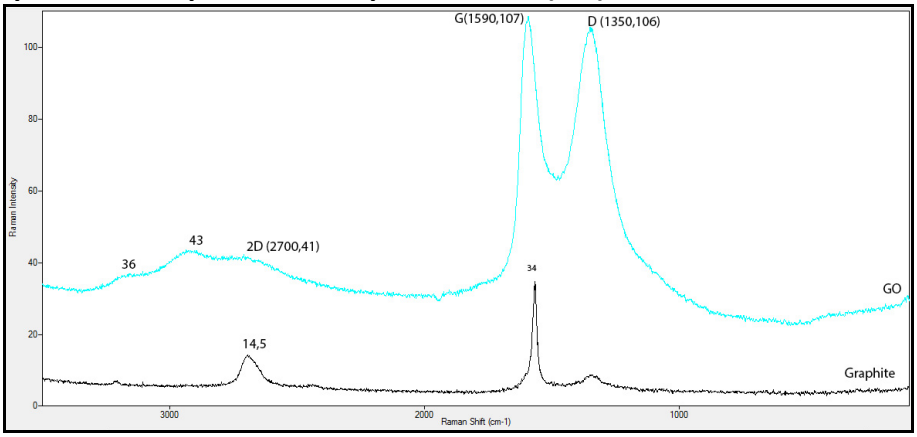


Fig. 6 Raman DXR Spectra of Graphite and GO

RAMAN DXR spectra of graphite and GO sample is given in Fig. 6. D peak (1350 cm^{-1}), G peak (1590 cm^{-1}), and 2D peak (2700 cm^{-1}) were indicated in Fig. 6. I_{2D}/I_G =0,38. This ratio corresponds nearly six layers GO structure.

The definition of layer numbers of obtained GO samples were performed by RAMAN, AFM, and TEM analysis. In this work, the same methods were used to determine the layer numbers of GO samples.

GO is a graphene based structure where Raman spectroscopy can be utilized as a characterization tool. The G mode in GO samples is wider and blue-shifted to ca. 1590 cm^{-1}, the D mode is significantly more intense (sometimes, even stronger than G mode) due to the disorder in the sp^2 structure induced by oxidative synthesis of GO and also due to the attachment of hydroxyl and epoxide groups on the carbon basal plane. The 2D mode with respect to the D and G modes is very small, but it can be enhanced after temperature annealing of GO which also affects the frequency position. It can shift by around 6 cm^{-1} and is related to the reduction of GO

AFM Analysis of Graphene Oxide (GO)

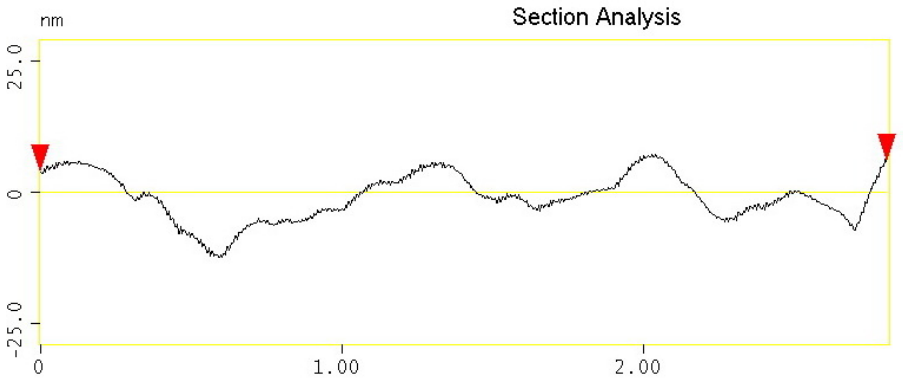


Fig. 7 AFM height profile of GO.

AFM analysis, performed for the determination of layer numbers of GO sample, was shown in Fig. 6. According to the height profile, vertical distance of scanned direction was 1.88 nm. The distance between graphene layers was known as 3.35 °A theoretically. Our GO sample has 0,782nm distance between layers. Thus, it can be concluded that our GO sample has 3-6 layers.

BET Analysis of Graphene Oxide (GO)

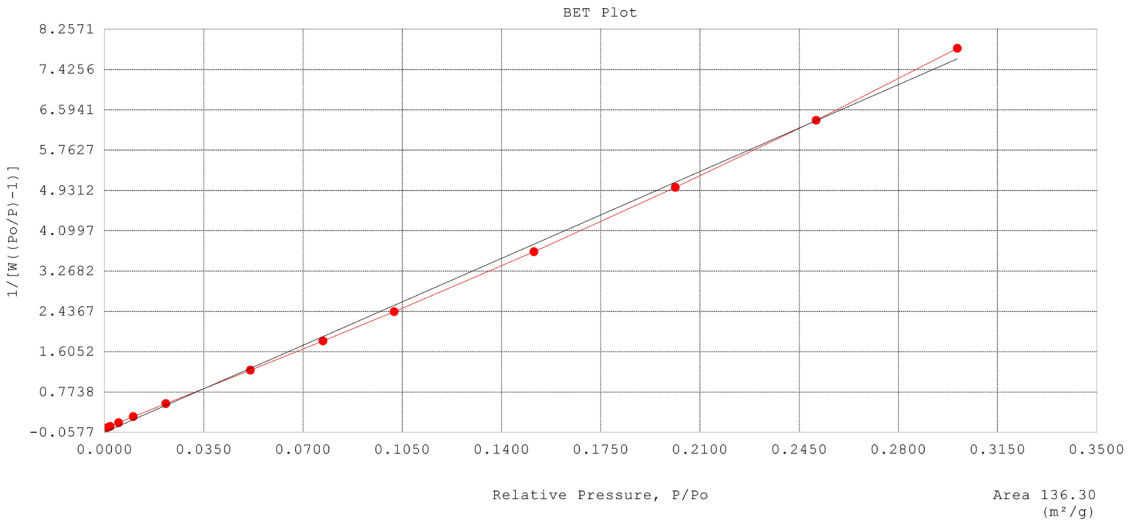


Fig. 8 BET plot of GO.

Specific surface area for GO is obtained from BET analysis as 136.3 m^2/gr.

TEM Images of Graphene Oxide (GO)

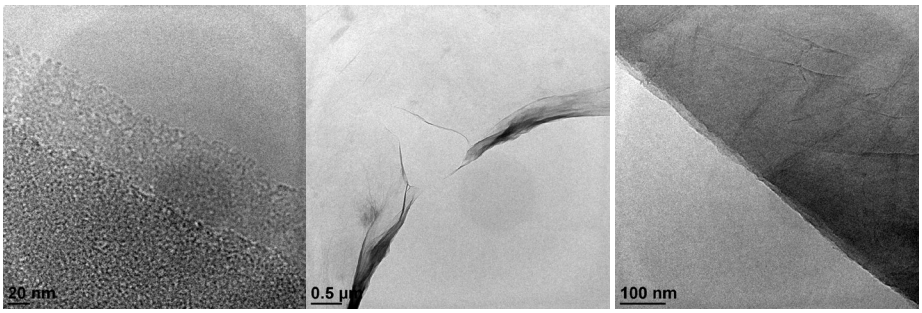


Fig. 9 TEM images of GO

TEM images of GO are shown in Fig.9 TEM images indicates some few layered GO edge formations.

Transformation of graphite to GO was characterized by XRD analysis. (2θ=10.88 characteristic peak of GO). The oxidation degrees calculated by C:O ratios of GO samples were obtained from EDX analysis. C:O ratio is 1.55. The functional groups were defined by FT-IR analysis. RAMAN, AFM, and TEM spectroscopies were used to define layer numbers of GO samples. Consequently, it was concluded that the synthesized GO has 3-6 layers.

REDUCED GRAPHENE OXIDE (RGO)

X-Ray Diffraction Pattern of Graphene Oxide (GO) and Reduced Graphene Oxide (RGO)

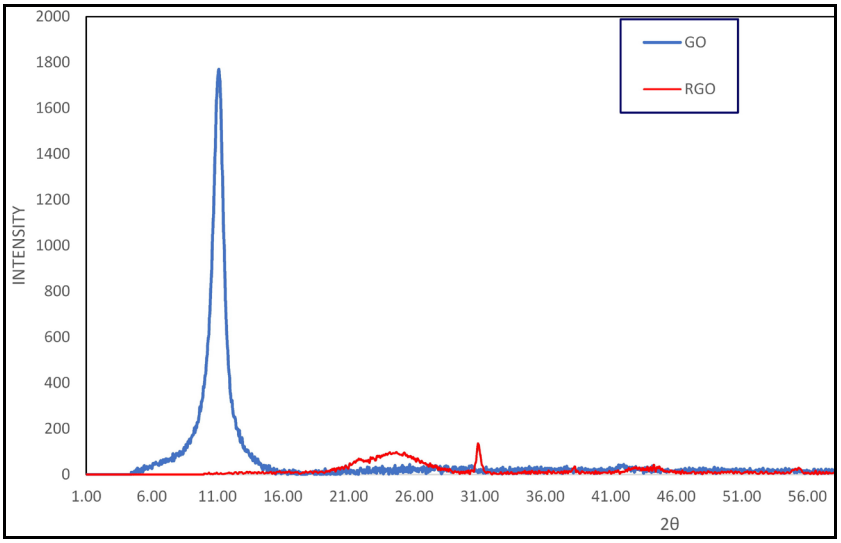
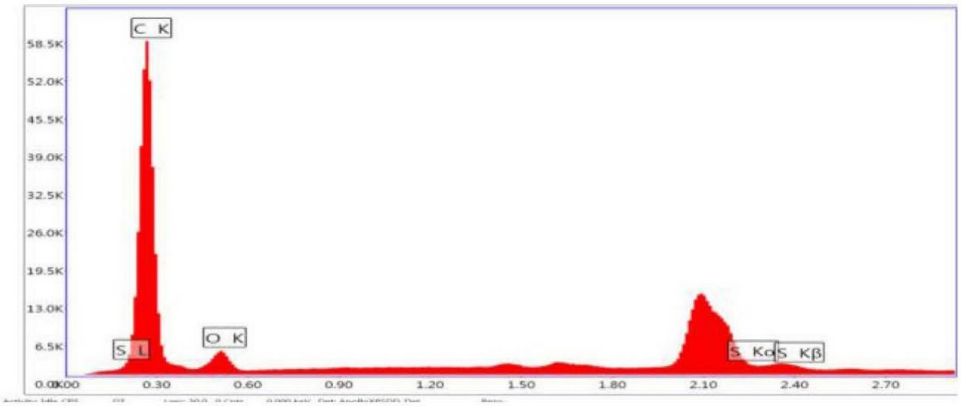


Fig. 10 XRD Pattern of GO and RGO

Graphite flakes exhibits a strong and sharp peak at 26.48° indicating a higher ordered structure, that corresponds to a basal spacing d_{002} = 0.334nm. The pattern of graphene oxide, on the other hand, exhibits a 001 reflection at 11.20 corresponding to a basal spacing of d_{001} = 0. 782nm.This value is higher than interlayer spacing of graphite flakes (d-spacing= 0.334nm, 2Θ =26.4°), due to the presence of oxygenated functional groups and intercalated water molecules. XRD patterns of GO is given in Fig. 1. GO shows characteristic GO peak between 2θ=9-11°. As a result of reduction, the characteristic peak of GO. RGO's XRD pattern shows its indicator at 2θ=27-30°.

EDX-EDS Analysis of RGO



Element	Weight %	Atomic	Net Int.	Net Int. Error
C K	81.63	88.26	10529.73	0
O K	10.59	8.59	975.56	0.01

Figure 11. EDS-EDX analysis of RGO

The atomic C:O ratio of RGO derived from EDX analysis is >8. The reduction degree of RGO was calculated by using C:O ratio.

SEM Images of RGO

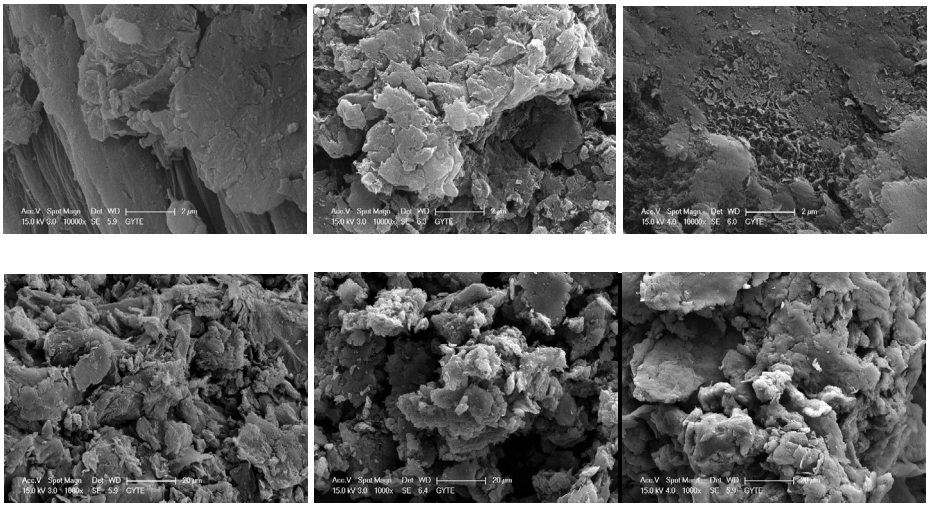


Figure 12. SEM Images of RGO

FTIR Spectra of RGO

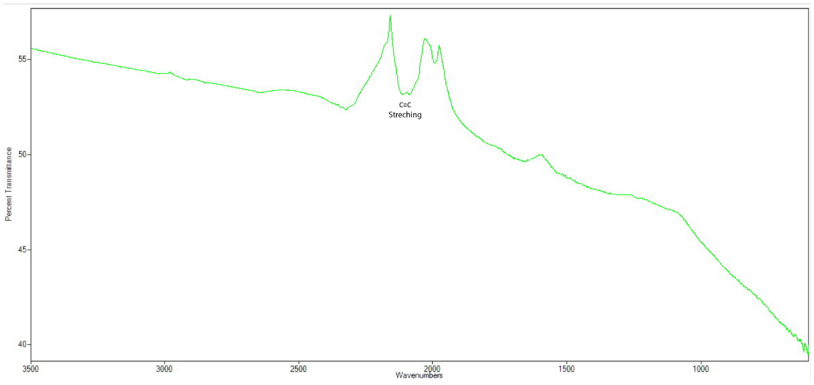


Figure 13. FTIR Spectrum of RGO

FTIR spectrum of RGO shows only C≡C stretching bond that indicates removal of oxygen functional groups.

DXR Raman Spectrum RGO

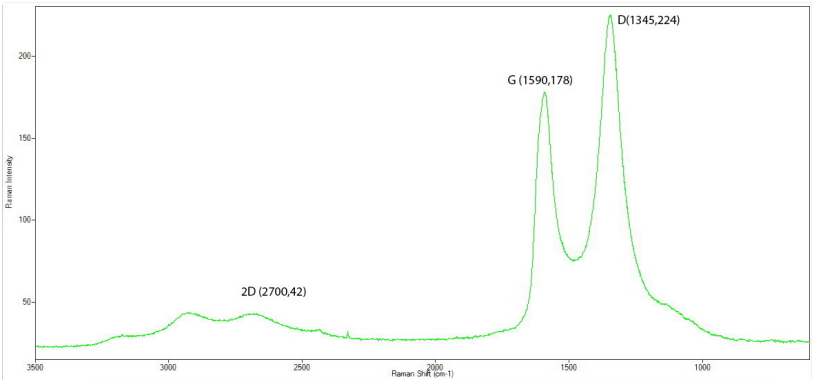


Figure 14. DXR Raman Spectrum of RGO

RAMAN DXR spectrum of RGO sample is given in Fig. 5. D peak (1345 cm^{-1}), G peak (1590 cm^{-1}), and 2D peak (2700 cm^{-1}) were indicated in Fig. 5. I_{2D}/I_G =0,19. This ratio corresponds 3-6 layers RGO structure.

TGA Analysis of RGO

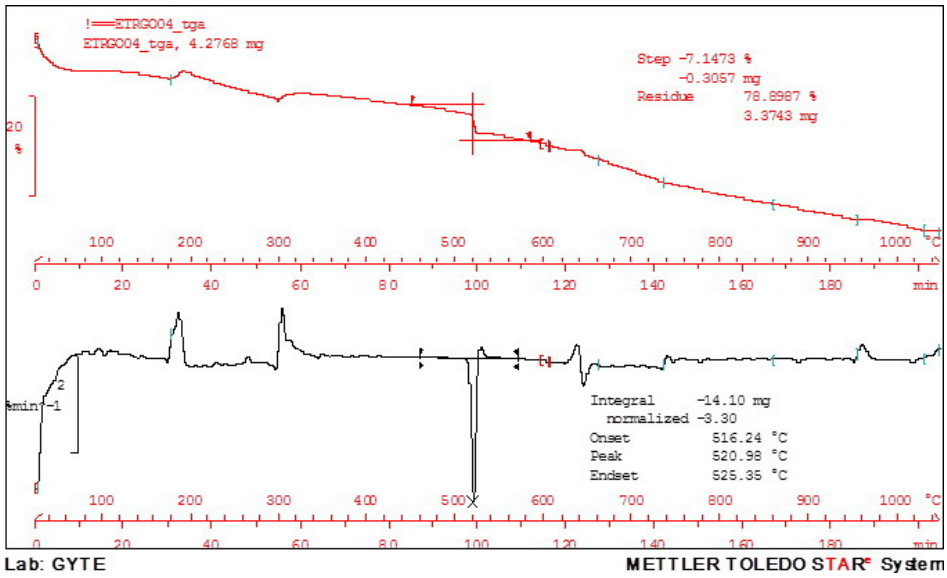


Figure 15. TGA Analysis of RGO

The removal of the thermally labile oxygen functional groups by chemical reduction results in much increased thermal stability for the reduced GO. Apart from a slight mass loss below 100 °C, which can be attributed to the loss of adsorbed water, no significant mass loss is detected when this material is heated up to 1000 °C.

AFM Analysis of GO

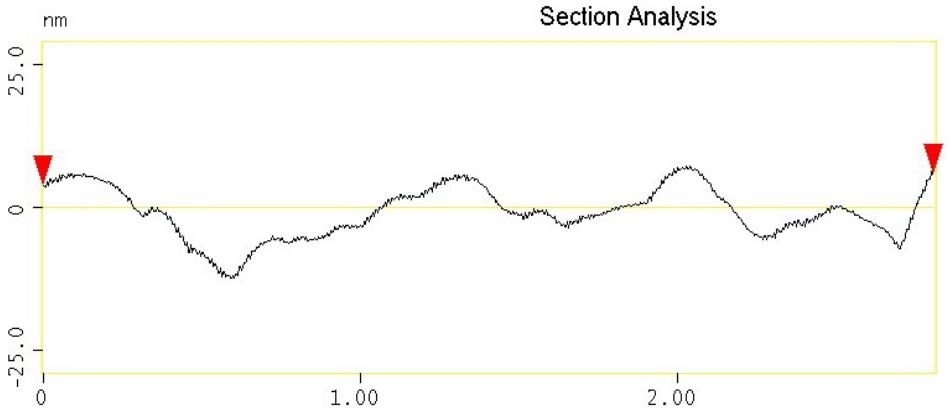


Fig. 16. AFM height profile of RGO.

AFM analysis, performed for the determination of layer numbers of GO sample, was shown in Fig. 16. According to the height profile, vertical distance of scanned direction was altered from 0.85mm to 1.88 nm. The distance between graphene layers was known as 3.35 °A theoretically. Our GO sample has 0,782nm distance between layers. Thus, it can be concluded that our RGO sample has 3-6 layers.

BET Analysis of RGO

Specific surface area for ETRGO05 is obtained from BET analysis as 657.46 $m^{2/}gr$.

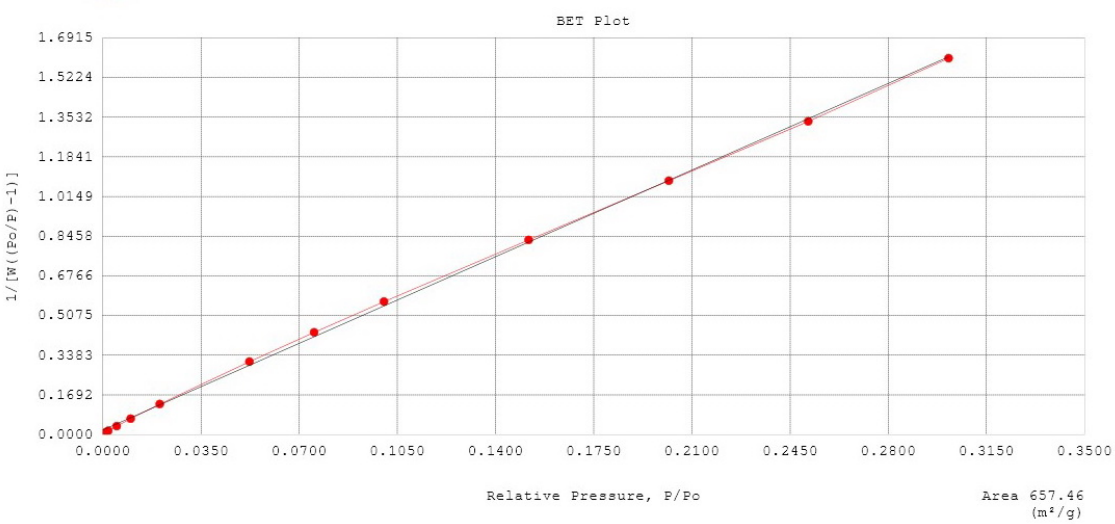


Figure 17. BET plot of RGO

TEM Images of RGO

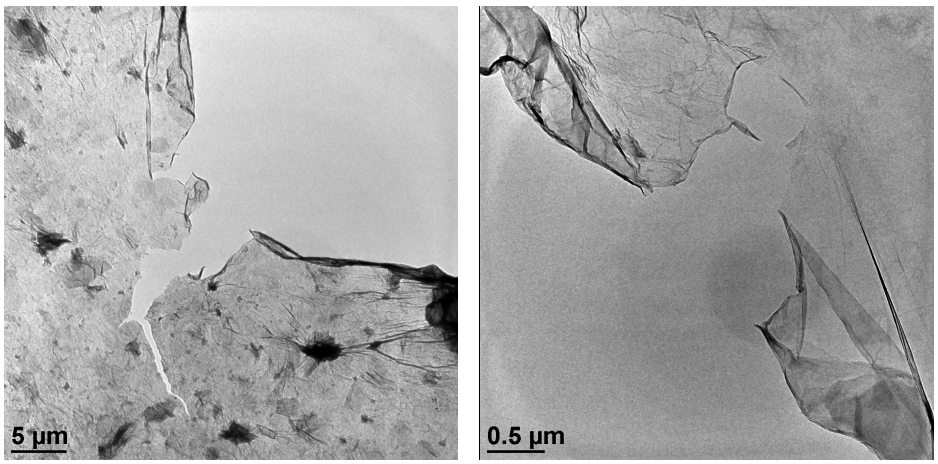


Figure 18. TEM images of RGO

TEM images of RGO are shown in Fig.17 TEM images indicates some few layered RGO edge formations.

Transformation of GO to RGO was characterized by XRD analysis. (2θ=10.88 characteristic peak of GO disappeared and RGO indicator peak appeared at 2θ=27-30 °). The reduction degree calculated by C:O ratios of RGO samples were obtained from EDX analysis. C:O ratio is >8. The functional groups were defined by FT-IR analysis. BET analysis gives 657.46 $m^{2/}gr$ specific surface area for RGO. TEM images shows few layered RGO structure. RAMAN analysis indicates 3-6 layered RGO structure. Consequently, it was concluded that the synthesized RGO has 3-6 layers.

CONTACT

Hazerfen

Kimya Malzeme ve Enerji Teknolojileri Sanayi Ticaret A.Ş.

Address
Cumhuriyet Mah. 2254.Sk. Gebze Teknik Üniversitesi No:2 G.Y.T.E., Teknoloji Transfer Ofisi, P. K. 41400

GEBZE, KOCAELİ, TÜRKİYE

Phone : +90 262 653 48 49
E-mail : info@hazerfen.com.tr
Web : www.hazerfen.com.tr