

ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В. Сокольский атындағы
«Жанармай, катализ және электрохимия институты» АҚ

Х А Б А Р Л А Р Ы

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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
электрохимии им. Д.В. Сокольского»

N E W S

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
JSC «D.V. Sokolsky institute of fuel,
catalysis and electrochemistry»

SERIES
CHEMISTRY AND TECHNOLOGY
4 (453)

OCTOBER – DECEMBER 2022

PUBLISHED SINCE JANUARY 1947

PUBLISHED 4 TIMES A YEAR

ALMATY, NAS RK

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«ҚР ҰҒА Хабарлары. Химия және технология сериясы»

ISSN 2518-1491 (Online),

ISSN 2224-5286 (Print)

Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.). Қазақстан Республикасының Ақпарат және қоғамдық даму министрлігінің Ақпарат комитетінде 29.07.2020 ж. берілген № **KZ66VPY00025419** мерзімдік басылым тіркеуіне қойылу туралы куәлік.

Тақырыптық бағыты: *органикалық химия, бейорганикалық химия, катализ, электрохимия және коррозия, фармацевтикалық химия және технологиялар.*

Мерзімділігі: жылына 4 рет.

Тиражы: 300 дана.

Редакцияның мекен-жайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., тел.: 272-13-19

<http://chemistry-technology.kz/index.php/en/arithv>

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Редакцияның мекенжайы: 050100, Алматы қ., Қонаев к-сі, 142, «Д.В. Сокольский атындағы отын, катализ және электрохимия институты» АҚ, каб. 310, тел. 291-62-80, факс 291-57-22, e-mail: orgcat@nursat.kz

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«Известия НАН РК. Серия химии и технологий».

ISSN 2518-1491 (Online),

ISSN 2224-5286 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).

Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан № KZ66VPY00025419, выданное 29.07.2020 г.

Тематическая направленность: *органическая химия, неорганическая химия, катализ, электрохимия и коррозия, фармацевтическая химия и технологии.*

Периодичность: 4 раз в год.

Тираж: 300 экземпляров.

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, оф. 219, тел.: 272-13-19

<http://chemistry-technology.kz/index.php/en/archiv>

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Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75.

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News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.

ISSN 2518-1491 (Online),

ISSN 2224-5286 (Print)

Owner: RPA «National Academy of Sciences of the Republic of Kazakhstan» (Almaty).

The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan No. **KZ66VPY00025419**, issued 29.07.2020.

Thematic scope: *organic chemistry, inorganic chemistry, catalysis, electrochemistry and corrosion, pharmaceutical chemistry and technology.*

Periodicity: 4 times a year.

Circulation: 300 copies.

Editorial address: 28, Shevchenko str., of. 219, Almaty, 050010, tel. 272-13-19

<http://chemistry-technology.kz/index.php/en/arhiv>

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Editorial address: JSC «D.V. Sokolsky institute of fuel, catalysis and electrochemistry», 142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22, e-mail: orgcat@nursat.kz

Address of printing house: ST «Aruna», 75, Muratbayev str, Almaty.

NEWS

OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC
OF KAZAKHSTAN

SERIES CHEMISTRY AND TECHNOLOGY

<https://doi.org/10.32014/2518-1491.135>

Volume 4, Number 453 (2022) 69-79

UDC 544.7, 543.4

IRSTI 31.15.35

**R. Kudaibergenova^{2*}, N. Murzakassymova², S. Kantarbaeva², D. Altynbekova²,
G. Sugurbekova¹**

¹Leading Researcher, Ecostandart.kz LLP, Astana, Kazakhstan;

²Taraz Regional University named after M.Kh. Dulaty, Taraz, Kazakhstan.

E-mail: rabi_07@bk.ru

RAMAN SPECTROSCOPY OF GRAPHENE, GO, RGO

Abstract. Graphene is the main focus of research in many modern fields of science. Graphene and its derivatives have excellent electronic, optical and mechanical properties, which have aroused great scientific interest and potential for various applications. Raman spectroscopy is one of the primary methods for characterizing graphene materials, as it has significant advantages over other analytical methods. Raman spectroscopy makes it possible to understand the quality of measured grapheme, including its structure and conductivity and the presence of impurities. Also, it is a universal tool for the determination and characterization of chemical and physical properties of these materials in laboratory conditions and on the scale of mass production. The importance of this method is such that most published articles on graphene studies have at least one Raman spectrum. Therefore, we systematically consider the Raman spectroscopy of graphene, graphene oxide (GO) and reduced graphene oxide (RGO) in this work. We describe the essential processes of Raman scattering of all modes of the first and second order in graphene, GO and RGO. In addition, shifts with different accumulation regimes, multilayer respiration, G and 2D multilayer graphene modes are discussed. Methods for determining the number of graphene layers, studying the Raman resonance spectra of single-layer and multilayer graphene and obtaining Raman images of graphene-based materials are proposed. The vast possibilities of Raman spectroscopy for the study of the fundamental properties of graphene are described.

Key words: graphene, graphene oxide, reduced graphene oxide, Raman spectroscopy.

**Р.М. Кудайбергенова^{2*}, Н.С. Мурзакасымова², С.М. Кантарбаева²,
Д.Т. Алтынбекова², Г.К. Сугурбекова¹**

¹Экостандарт.kz ЖШС, Қазақстан, Астана, Қазақстан;

² М.Х.Дулати атындағы Тараз өңірлік университеті, Тараз, Қазақстан.
E-mail:rabi_07@bk.ru

ГРАФЕН, ГО, ТГО РАМАНДЫҚ СПЕКТРОСКОПИЯСЫ

Аннотация. Графен көптеген заманауи ғылым салаларындағы негізгі зерттеу нысандырының бірі болып табылады. Графен және оның туындылары тамаша электрондық, оптикалық және механикалық қасиеттерге ие болғандықтан, олар үлкен ғылыми қызығушылық пен әртүрлі қолданыстарда жоғары сұраныс тудырды. Раман спектроскопиясы графен негізіндегі материалдарды зерттеудің негізгі әдістерінің бірі болып табылады, өйткені оның басқа аналитикалық әдістерге қарағанда айтарлықтай артықшылықтары бар. Олардың ең маңыздысы үлгіні дайындаудың қарапайымдылығы және алынған ақпараттың үлкен көлемі. Раман спектроскопиясы графеннің сапасын, тазалығын және оның құрылымы мен өткізгіштігін, сондай-ақ қоспалардың болуын талдауға мүмкіндік береді. Сонымен қатар, зертханалық және өндіріс жағдайларда графен негізіндегі материалдардың химиялық және физикалық қасиеттерін анықтауға және сипаттауға арналған әмбебап құрал. Бұл талдау әдісінің маңыздылығы сонша, графенді зерттеу бойынша жарияланған мақалалардың көпшілігінде кем дегенде бір Раман спектрі бар. Сондықтан, бұл жұмыста біз графеннің, графен оксидінің (ГО) және тотықсызданған графен оксидінің (ТГО) Раман спектроскопиясын жүйелі түрде қарастырамыз. Графенде, ГО және ТГО-де бірінші және екінші ретті режимдер сәулесінің Рамандық шашырауының маңызды процестері сипатталған. Сонымен қатар, әртүрлі жинақтау режимдері бар жылжулар, көп қабатты графеннің G және 2D режимдері талқыланады. Сондай-ақ графен қабаттарының санын анықтау, бір қабатты және көп қабатты графеннің Раман резонанстық спектрлерін зерттеу және графен негізіндегі материалдардың Раман кескіндерін алу әдістері ұсынылған. Графеннің іргелі қасиеттерін зерттеуге арналған Раман спектроскопия әдісінің кең мүмкіндіктері сипатталған.

Түйін сөздер: графен, графен оксиді, тотықсызданған графен оксиді, Раман спектроскопиясы.

Р.М. Кудайбергенова^{2*}, Н.С. Мурзакасымова², С.М. Кантарбаева²,
Д.Т. Алтынбекова², Г.К. Сугурбекова¹

¹ТОО «Экостандарт.kz», Астана, Казахстан;

² Таразский региональный университет имени М.Х. Дулати, Тараз, Казахстан.
E-mail:rabi_07@bk.ru

РАМАНОВСКАЯ СПЕКТРОСКОПИЯ ГРАФЕНА, ГО, ВГО

Аннотация. Графен является основным объектом исследований во многих современных областях науки. Графен и его производные обладают отличными электронными, оптическими и механическими свойствами, которые вызвали большой научный интерес и большой потенциал для различных приложений. Рамановская спектроскопия является одним из основных методов исследования графеновых материалов, так как имеет значительные преимущества перед другими аналитическими методами. Наиболее важными из них являются простота пробоподготовки и большой объем получаемой информации. Рамановская спектроскопия позволяет понять чистоту и качество измеренного графена, включая его структуру и проводимость, а также наличие примесей. Также она является универсальным средством для определения и характеристики химических и физических свойств этих материалов как в лабораторных условиях, так и в масштабах серийного производства. Важность этого метода такова, что в большинстве опубликованных статей по исследованию графена есть по крайней мере один спектр комбинационного рассеяния. В статье рассматривается рамановская спектроскопия графена, оксида графена (ГО) и восстановленного оксида графена (ВГО). Описаны важные процессы комбинационного рассеяния света всех мод первого и второго порядка в графене, ГО и ВГО. Кроме того, обсуждаются сдвиги с разными режимами накопления, многослойное дыхание, режимы G и 2D многослойного графена. Предложены также методы определения количества слоев графена, исследования спектров рамановского резонанса однослойного и многослойного графена и получения рамановских изображений материалов на основе графена. Описаны широкие возможности рамановской спектроскопии для изучения фундаментальных свойств графена.

Ключевые слова: графен, оксид графена, восстановленный оксид графена, рамановская спектроскопия.

Introduction. Graphene is a two-dimensional (2d) hexagonal system of sp² carbon atoms with solid covalent bonds (Dreyer et al., 2010). Forming the van-der-Waals bonds graphene can be layered in Bernal or rhombohedral form (Nicolosi et al., 2013). Single-layer and multilayer graphene can be obtained by micromechanical exfoliation (Kumar et al., 2021), chemical vapor deposition (CVD) (Fidanova et al., 2019) and epitaxial growth on the SiC surface (Yazdi et al., 2016), and there are methods such as reduction of GO, organic synthesis, and subjecting graphene to oxidation, hydrogenation,

fluorination, and other chemical functionalizations (Stankovitch et al., 2007). Graphene and its derivatives have excellent electronic, optical and mechanical properties, which have aroused great scientific interest and potential for various applications (Jariwala et al., 2013).

In recent years, interest in studying the fundamental properties of graphene has grown due to the simplicity of fabrication and identification of structures (Kudaibergenova et al., 2020). Raman spectroscopy is a relatively simple, non-destructive, non-contact and fast measurement method for studying the inelastic scattering of light from a sample surface at room temperature and atmospheric pressure (Scardaciet al., 2021). Raman spectroscopy plays an essential role in the structural description of materials based on graphene. Furthermore, it is an indispensable tool for understanding the actions of electrons and phonons in graphene, which we can obtain by studying Raman spectroscopy (Ferrari et al., 2013).

Raman spectroscopy has significant advantages over other analytical methods. The most important of them are the ease of sample preparation and the large amount of information obtained. Raman spectroscopy is a technique based on light scattering, so all that is required to collect a spectrum is to point the incident beam exactly at the sample and then collect the scattered light (Hidayahet al., 2017).

The purpose of this work is to study the exact identification of graphene and its functional groups using the Raman spectrum method. Because vibrational spectroscopy provides key information about the structure of molecules, such as the position and intensity of bands in a spectrum, it can be used to study the molecular structure or chemical identification of a sample.

Materials and Methods. Chemicals and apparatus. Graphite (purity $\geq 99\%$), sulfuric acid (H_2SO_4), sodium nitrate (NaNO_3), potassium permanganate (purity $\geq 97\%$), hydrazine monohydrate ($\text{NH}_2\text{-NH}_2\cdot\text{H}_2\text{O}$, purity =98%) were purchased from Sigma-Aldrich.

Graphite, GO and RGO were described using the method Raman spectroscopy. The measurements were performed using an Omegascope Horiba-AIST-NT system (with a Raman spectrometer LabRAM HR Evolution) with a 532 nm laser.

Preparation of GO and RGO. Synthesis of GO was performed according to the modified Hummers method (Hummers et al., 1958), as described in (Kudaibergenova et al., 2022). The resulting GO solution was washed with HCl and water at pH 1.1, then centrifuged and dried to constant weight.

The reduction of GO is described in (Kudaibergenova et al., 2022). $\text{NH}_2\text{-NH}_2\cdot\text{H}_2\text{O}$ was added to the colloidal suspension of GO (figure 1). The resulting mixture was stirred, filtered and dried.

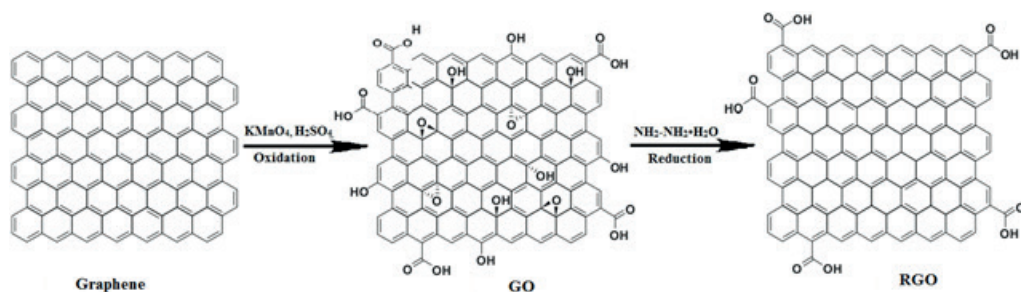


Figure 1. Schematic image of the synthesis of graphene, GO and RGO.

Results and discussion. Raman spectra of graphite contain bands G (graphite) and D (disorder). The synthesized GO sheets using the modified Hummers and RGO sheets also are characterized by Raman spectroscopy (Figure1). It is known (Wuet al., 2018) that three peaks are present in a typical Raman spectrum of graphene sheets: the first peak D at 1351 cm⁻¹, the second peak G at 1580 cm⁻¹, and the third peak 2D at 2700 cm⁻¹. The ratio between the intensity of the G and 2D peaks (IG/I2D) estimates the number of layers (Popov et al., 2013).

This IG/I2D ratio is less than unity. The relationship between the D and G peaks (ID/IG) assesses the imperfection of graphene layers. The prominent Raman signal at 1351 cm⁻¹ is attributed to the sp³ defects in the sp² lattice and related to the graphene structure defects. A 2D peak at 2700 cm⁻¹ is attributed to the development of graphene structure. In the Raman spectrum of RGO, the D, G, and 2D peaks appear at 1358, 1589 and 2709 cm⁻¹. These peaks are associated with the known peaks for GO found in the literature, and the obtained graphene sheets show strongly broadened D and G bands. The intensity of the D peak increases after reduction (Hidayahet al., 2017). The most noticeable in the Raman spectra of graphite materials are the G band appearing in the region of 1582 cm⁻¹, the D band of 1350 cm⁻¹, the D band of 1620 cm⁻¹ and the G band (or 2D) of about 2700 cm⁻¹ (for these the last three bands, this combination frequency is equal to the frequency obtained using the wavelength of laser excitation at 514.5 nm). The G band is a doubly degenerate phonon mode of symmetry E_{2g} in the center of the Brillouin zone (BZ), which is a combination for sp² carbon networks. If the G band is observed in Raman spectra, which means that the sample contains sp² carbon networks. In contrast to sp³ and sp carbon networks show characteristic Raman features at 1333 cm⁻¹ and in the range of 1850–2100 cm⁻¹ (linear carbon chains) (Kaniyoor et al., 2012).

The D' band is associated with lattice defects, and therefore these bands are not visible for highly crystalline graphite (Figure2, Table 1). Thus, the ratio of the intensities ID / IG for the D-band and G-band widely characterises the number of defects in graphite materials.

The prominent characteristic peaks in the Raman spectra of graphene and graphite are G (~ 1580 cm⁻¹), D (~ 1330 cm⁻¹) and G' (~ 2670 cm⁻¹). The G band arises due to plane vibrations of sp² carbon atoms. Bands G, D and D' are caused by disorders resulting from exfoliation. Band D is associated with crystalline disease due to defects in the lattice, and band G' is the overtone of band D. Figure 2 shows Raman spectra of

graphite, graphene oxide (GO), and reduced graphene oxide (RGO). It can be observed that all three structures have defects (peak D at 1335 cm^{-1}). The presence of defects in graphene oxide due to violation of π -conjugation leads to GO becoming electrically nonconducting. Removal of functional groups leads to the restoration of π -conjugation and an increase in the material's conductivity (Kudaibergenova et al., 2022).

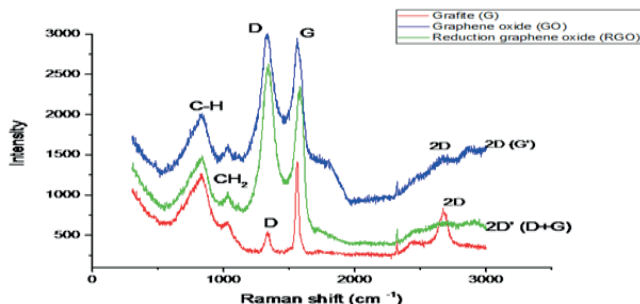


Figure 2. Raman spectrum of graphene, GO and RGO

Fluctuations in the frequency range 1500 cm^{-1} are due to the presence of microcrystallite fragments in the structure. For a qualitative comparison of particle defects, the I_D / I_G ratio can be used. However, the intensity of the D and G bands of the studied samples turned out to be commensurate, and the I_D values are slightly higher than the I_G values. This also indicates a rather large number of defects in the sample. $I_D / I_G = 1.03$ for GO and 1.11 for RGO (Table 1). For the graphite the value $I_D / I_G = 0,38$.

The G peak refers to tangential C-C stretching vibrations and is sensitive to stresses in the local structure. Stresses can occur due to bending. In places of greatest curvature, a decrease in the frequency of oscillations of the G mode is observed. Such a frequency shift corresponds to tensile strains. Additionally, at the site of the greatest curvature, an increase in the intensity of the D peak is observed. The D band also indicates the presence of defects in lattice imperfections or the presence of impurity atoms. More defects in the structure - a more intense peak. From the I_D / I_G ratio, the intensity of one or another type of defect is determined. If the ratio is <1 , then the defectiveness is low, and the quality of the carbon material is high. Graphite has a ratio $I_D / I_G = 0,38$.

Group G occurs during the planar vibration of sp^2 carbon atoms and is a doubly degenerate (TO and LO) phonon mode (E_{2g} symmetry) in the center of the Brillouin zone (Mallet-Ladeira et al., 2014).

In the Raman spectrum of graphite, the most intense narrow peak, G, is at 1563 cm^{-1} (Figure2, Table 1). In the case of graphite with a large number of defects, a peak D (disorder peak) appears at 1335 cm^{-1} in the spectrum. The I_D / I_G ratio is associated with the size of graphene crystallites in the basal plane, L_a (Tuinstra et al., 1970):

$$L_a = (2,4 \times 10^{-10}) \lambda^4_{\text{laser}} (I_D / I_G)^{-1},$$

where λ is the wavelength of the exciting laser, equal to 514 nm.

We used the “Tuinstra Koenig Relation” ratio method to calculate the crystallite size

using Raman spectroscopy. To calculate the number of layers of the obtained materials, we used the practice of the ratio of the intensity of peaks D: G in the Raman spectra (Table 1) (Tuinstra et al., 1970).

Determination of the thickness of graphene films is one of the most critical tasks in their diagnosis since the properties of graphene substantially depend on the number of its layers. Table 1 shows that with an increase in the number of layers, the intensity of the G line increases while the 2D line broadens and changes its shape. Therefore, an analysis of the 2D line shape in the Raman spectrum of graphene and an analysis of the G-line intensity are used to estimate its thickness (Kaniyoor et al., 2012).

According to table 1, the intensity of the band G increases with the increasing thickness of graphene oxide. These are used in the work of A. Geim to determine the thickness of graphene materials with less than 10 layers for two-dimensional materials (Geimet et al., 2007).

In the frequency range $\sim 2870\text{ cm}^{-1}$, a G' band appears in the Raman spectrum of GO (Figure 2 and Table 1). The Raman spectrum of graphene oxide differs from the graphite spectrum in the intensity of the G'-band ($\sim 2870\text{ cm}^{-1}$). It is because identifying the number of layers in the graphene structure is carried out on the G'-band. In some cases, instead of band D, you can use band G' if the I_D / I_G ratio is > 1 .

The broader and less intense G' band at 2708 cm^{-1} (sometimes called the 2D peak in the literature) in the RGO spectrum compared to the graphite peak at 2673 cm^{-1} suggests that the RGO sheets contain more defects than graphite, which also indicates the ratio $I_D / I_G = 1.11$ (table 1). A wide 2D peak for GO was observed at 2670 cm^{-1} , but with a significant increase in peak intensity (Figure 2, Table 1). This fact confirms the increase in the thickness and number of defects in GO due to chemical exfoliation of graphite during oxidation. For single-layer graphene, the G' band appears as a sharp and symmetric peak, while it becomes wider with increasing thickness in several-layer graphene (Kudaibergenova et al., 2022).

For example, the single-layer graphene (SLG) of a 2D band has a narrow and symmetric peak. Figure 2 shows that the 2D peak for GO and RGO becomes wider and more intense, i.e., with the chemical modification of graphite and reduction, the number of layers in the material structure increases. Authors (Schuepferet al. 2020) explained the nature of the expansion of the 2D band. Also gave a detailed description of the fact that the multi-peak structure of the 2D band is associated with the dispersion of π electrons. The intensity of the 2D band can be used to distinguish a monolayer from 5-10 stacked layers. The relative intensity ratio between the 2D and G bands depends on the number of layers. I_{2D} / I_G can be used to understand the quality of graphene samples quickly. (Camposet al. 2017) discussed that the bandwidth D is sensitive to the effects of the curvature of graphene sheets, as well as some morphological changes, which are also accompanied by a change in the Raman spectrum.

In the Raman spectra of the materials we obtained, there is a wide line at 826 cm^{-1} for graphite and an intense band at 833 cm^{-1} for GO and 835 cm^{-1} for RGO (Figure 2), which is shifted to the high-frequency region by 53 cm^{-1} compared with a band at 782 cm^{-1} in the works, referred to C – H bending vibration of the ring (Camposet al. 2017).

Table 1. Analysis of Raman spectra of graphite, GO и RGO

Sample	Raman shift (cm ⁻¹)	Intensity	Peak identification	I _D /I _G	I _{2D} /I _G	Crystallite size L _a , (nm)	I _D /I _{D'}
Graphite	1563	1420	G	0.38	0.61	44.08	-
	1335	540	D				
	1030	670	C-H				
	826	1260	CH ₂				
	2440	430	D+D''				
	2673	870	2D				
GO	1561	2950	G	1.03	0.51	16.26	2.1
	1337	3040	D				
	1040	1620	C-H				
	833	2000	CH ₂				
	1800	1450	D'(light scattering on the linear defects)				
	2670	1500	2D				
	2870	1600	G'				
2992	1620	2D' (D+G)					
RGO	1583	2350	G	1.11	0.29	15.09	-
	1341	2600	D				
	1040	1040	C-H				
	835	1480	CH ₂				
	2708	680	2D				
	2946	700	2D' (D+G)				

The lines located in the regions of 1031 and 1073 cm⁻¹ were assigned to the C – C stretching associated with the respiration of the phenyl ring and possibly belong to the vibrations of the side groups (Kudaibergenova et al., 2022). The line at 1030 cm⁻¹ in the Raman spectrum of graphite was assigned to plane vibrations of the CH₂ groups of the ring. The line located at 1040 cm⁻¹ in the Raman spectrum of GO и RGO is also due to stretching processes associated with this line with C-C antisymmetric stretching along the macromolecular chain. The position of this Raman line shifts toward lower values. In our case, this band is present in the region of 1040 cm⁻¹ for GO and RGO (Figure 2, Table 1).

In work (Kudaibergenova et al., 2022), it was reported that the D line in graphene at 1336.1 cm⁻¹ indicates that the graphene plate consists of 3-5 layers and can reflect compression deformation, crowding, or cohesion of the side groups. We are inclined to assume that the D peak in our Raman spectra of graphite at 1335 cm⁻¹ and the high-intensity peaks at 1336.5 cm⁻¹ - GO and 1341 cm⁻¹ - RGO also reflect similar phenomena (Figure 2, table 1).

The 2D peak is the overtone of the D peak, and the 2D' peak is the overtone of the D' peak. 2D and 2D' peaks are activated only if there are defects. The band at ~ 2440 cm⁻¹, presented only in the Raman spectrum of GO in Figure 1 we interpreted as D + D'' (Table 1). The D + D'' band was first represented in graphite. This band is a combination of a D-phonon and a phonon belonging to the LA branch, visible at ~ 1100 cm⁻¹ in defective

samples when measured by visible light and called the D'' peak and is denoted as D + D'' (Mallet-Ladeira et al., 2014).

Thus, we have revealed the origin of the two-resonance two-phonon D + D'' peak, which can provide useful information for functionalization in determining the number of layers in graphene (Schuepferet al. 2020). Raman peak at 2946 cm⁻¹ in the RGO spectrum in Figure 2 is associated with the D + G combination mode, and is also caused by the disorder of the structure and is often referred to in the literature as 2D' (Kaniyoor et al., 2012). Raman spectra show the structural characteristics of the material and information about defects, stacking of graphene layers and final crystallite sizes. Raman peaks D and D' are characterized by disorder, and the G' band (Overtone of the D band), is always observed in samples without defects (Hidayah et al., 2017). The identification of these bands is necessary for understanding the mechanism of the formation of new forms of sp² carbon materials. The most obvious difference in the intensity of the G' band (or 2D-band) in 2700 cm⁻¹, which in graphene exceeds the G band in intensity, and in graphite, this ratio reverses (Kudaibergenova et al., 2022).

Another example of the high sensitivity of the Raman spectrum to the structure of materials with lattice distortion at the crystal edge is the appearance of a rather intense shoulder at 1800 cm⁻¹ in the Raman spectrum of GO, which indicates the presence of carbon in the sp² state and the existence of linear defects at the crystal boundary (Hidayah et al., 2017). We found that the ratio of the peak intensities of the D and D' line intensities caused by the appearance of sp³ hybridization is ID / ID' = 2.1 (Table 1), which indicates the presence of linear defects at the GO boundary.

GO reduction was carried out using hydrazine, which led to a change in the structure of the material, which Raman spectra of graphite, GO and RGO (Figure 2).

The G peak corresponds to the high-frequency phonon E_{2g}. The D peak is due to the vibrational modes of the six-atom rings and refers to the A_{1g} symmetry. The intense peak G, which relates to vibrations of the graphite lattice, showed a high-frequency shift of the G band in the Raman spectrum of RGO (1583 cm⁻¹) compared with the G position of the graphite band (1562 cm⁻¹) and GO (1561 cm⁻¹), which indicates an increase in the concentration of free charge carriers (Kudaibergenova et al., 2022).

The blue shift of the G peak in the spectrum compared with the G band of graphite (1562 cm⁻¹) and GO (1561 cm⁻¹) increases the phonon energy E_{2g}. The increase in the frequency of the G line in the RGO spectrum (1583 cm⁻¹, Table 1) with increasing electron concentration is explained by the fact that the more electrons are in the π* band, the stronger is the contribution of the electron energy to the phonon energy E_{2g}. A quantitative expression for the change in the energy of the G line with a change in the carrier concentration was obtained in the work (Hidayah et al., 2017).

Conclusion. In this study, we described the synthesis of RGO in two stages, consisting of the preparation of GO from graphite by the modified Hummers method, followed by the chemical reduction of GO with hydrazine monohydrate. The location of the 2D band showed that the resulting GO appears to be multilayer, and also, the shifted position of the 2D band confirms the presence of functional oxide groups that interfere with the stacking of graphene layers. It was also found that after the reduction of GO to

RGO, a small amount of functional oxide groups remains, which leads to the binding of RGO. And the high intensity of the D band indicates that RGO has more isolated graphene domains than GO, and also due to the removal of oxygen fragments from GO after reduction. According to the results of Raman spectroscopy, it can be revealed that GO was successfully synthesized and chemically reduced using hydrazine hydrate.

Information about the authors:

Sugurbekova Gulnar – Doctor of Chemical Sciences, Leading Researcher, Ecostandart.kz LLP, Kabanbay batyr Ave 53, Astana, Kazakhstan, sugurbekova.g.55@gmail.com, <https://orcid.org/0000-0002-6894-7247>;

Kudaibergenova Rabiga – PhD doctoral student specializing in chemistry, Taraz Regional University named after M.Kh. Dulaty, st. Tole bi 60, Taraz, Kazakhstan, Tel.: +77476741776 rabi_07@bk.ru, <https://orcid.org/0000-0003-0759-1539>;

Murzakassymova Nazgul – master of chemistry, Senior teacher, department of «Chemistry and chemical technology», Taraz Regional University named after M.Kh. Dulaty, st. Tole bi 60, Taraz, Kazakhstan, <https://orcid.org/0000-0003-0329-6137>;

Kantarbaeva Saule – master of chemistry, Senior teacher, department of «Chemistry and chemical technology», Taraz Regional University named after M.Kh. Dulaty, meirhanovna@mail.ru, <https://orcid.org/0000-0001-5566-1672>;

Altynbekova Dinara – master of chemistry, Senior teacher, department of «Chemistry and chemical technology», Taraz Regional University named after M.Kh. Dulaty, st. Tole bi 60, Taraz, Kazakhstan, <https://orcid.org/0000-0002-8489-320X>.

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ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Директор отдела издания научных журналов НАН РК *А. Ботанқызы*
Заместитель директор отдела издания научных журналов НАН РК *Р. Жәлікқызы*

Редакторы: *М.С. Ахметова, Д.С. Аленов*

Верстка на компьютере *Г.Д. Жадырановой*

Подписано в печать 05.12.2022.

Формат 60x88¹/₈. Бумага офсетная. Печать – ризограф.

9,0 п.л. Тираж 300. Заказ 4.