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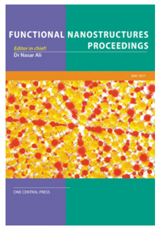

Development of a method of obtaining graphene layers from rice husk

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ABSTRACT

In this work, graphene layers were obtained from rice husk by potassium hydroxide activation followed by alkaline desilication. Rice husk samples was subjected to carbonization at the following conditions: 2 h of activation time, 850ºC and ratio of rice husk/KOH (wt/wt) was 1/4. Concentration of NaOH desilication solution was 1M. The obtained samples were studied using Raman spectroscopy, the peaks characterize the presence of graphene layers in the sample. The yield of the product was ~ 3% by weight.

I. INTRODUCTION

In the past years, there has been an interest on the investigation of two-dimensional carbon nanomaterials with atomic level thickness. As the top of this family, graphene has received considerable attention due to its unique electrical, mechanical and photothermal properties, and a variety of applications have been proposed in molecular devices, material and life sciences [1]. To realize these applications, scalable graphene production and subsequent sample engineering play a key role.

For several years, since the discovery of the first method for the obtaining of graphene based on the mechanical splitting of graphite layers, the efforts of many research laboratories have been aimed at developing new, more effective approaches to solving this problem. Many methods, such as liquid-phase separation of graphite, its oxidation, graphene synthesis by chemical vapor deposition, epitaxial growth of graphene on a metal surface, thermal decomposition of carbide, obtaining of graphene in an electric arc are used to obtain graphene [2,3]. However, these methods are very time-consuming, require a lot of time and have a low yield of graphene. Therefore, the search for new, more simple and cost-effective methods for the synthesis of graphene is an actual task. One such promising method can be the method of obtaining graphene layers from agricultural wastes. Agricultural wastes, e.g., rice husks, poplar tree, saxaul, corncob and apricot stones are usually decomposed by burning, which produces ash residues used as sorbents. Among these materials, rice husks have very good properties [4] and their stocks are huge in the Republic of Kazakhstan and other countries. In this regard assumed that the obtaining of graphene from rice husks opens the possibility of developing various applications due to its inexpensive, simple and scalable production.

II. MATERIALS AND METHODS

In this work, rice husk (RH), which is a multi-tonnage and renewable waste, was used as a raw material. KOH is used as a typical chemical reagent to induce porosity. This method of obtaining graphene oxide from rice husk was different from the one reported in [5-7]. The carbonized rice husk containing graphene was obtained in four successive stages: pre-carbonization, desilication, chemical activation and exfoliation of the carbonized rice husk (CRH).

Pre-carbonization of RH Firstly, the RH was washed several times with distilled water to remove impurities, then dried at 383 K for 1 hour. The method of pre-carbonization of RH was carried out in a rotating reactor in an inert medium at a temperature of 523-573 K, with an argon delivery rate of ~ 5 cm^3/min, the carbonization time was 45 minutes [8].

Desilication of CRH The resulting samples of CRH were desilted in 1M NaOH solution and heated at 353 K for 3 hours. The solution was then rinsed several times with distilled water to establish a neutral medium and dried at 383 K for 2 hours [9].

Chemical activation of CRH In this stage, the dried CRH was mixed with crushed KOH in a ratio of 1:4. The mixture was pressed in a refractory crucible and heated in a muffle furnace to a temperature of 1123 K for 2 hours. To prevent oxidation, argon was poured at a rate of 5 cm^3/min. The samples were washed several times with distilled water, then dried at 383 K for 2 hours.

Exfoliation of CRH To remove amorphous carbon, the exfoliation process of the CRH was carried out in a solution of hydrogen peroxide (H_2O_2, 37%) for 48 hours. The yield of the product was ~ 3% by weight.

III. RESULTS AND DISCUSSION

A quick and accurate way to determine the number of layers of graphene is of great importance in accelerating the study of this material. A previous work [10] demonstrated that the proposed method allows to obtain multilayer graphene. Therefore, in this work, Raman spectroscopy was used to determine the number of graphene layers. As is known, a typical spectrum of graphene exhibits three peaks: peak *D* at 1351 cm^{-1}, peak *G* at 1580 cm^{-1}, and peak *2D* at 2700 cm^{-1}. The ratio between the intensities of peak *G IG* and peak *2D* I_{2D}, I_G/I_{2D} gives an estimation of the number of layers. For monolayer graphene, this ratio is less than unity. The ratio between the intensities of peak *D* I_D and peak *G* I_G, I_D/I_G evaluates the defectiveness of graphene layers [11]. Figure 1a shows Raman spectrum of graphene layers obtained from CRH, the maximum number of graphene layers is less than ten (I_G/I_{2D} =2.06 and I_D/I_G =0.29), as indicated by the ratio between the peaks' intensities I_G/I_{2D}. It was shown [12] that the ratio of I_G/I_{2D} = 1.3 corresponds to three layers of graphene, whereas the authors of [13] found that I_G/I_{2D} = 1.8-2.4 corresponds to 5-10 layers of graphene.

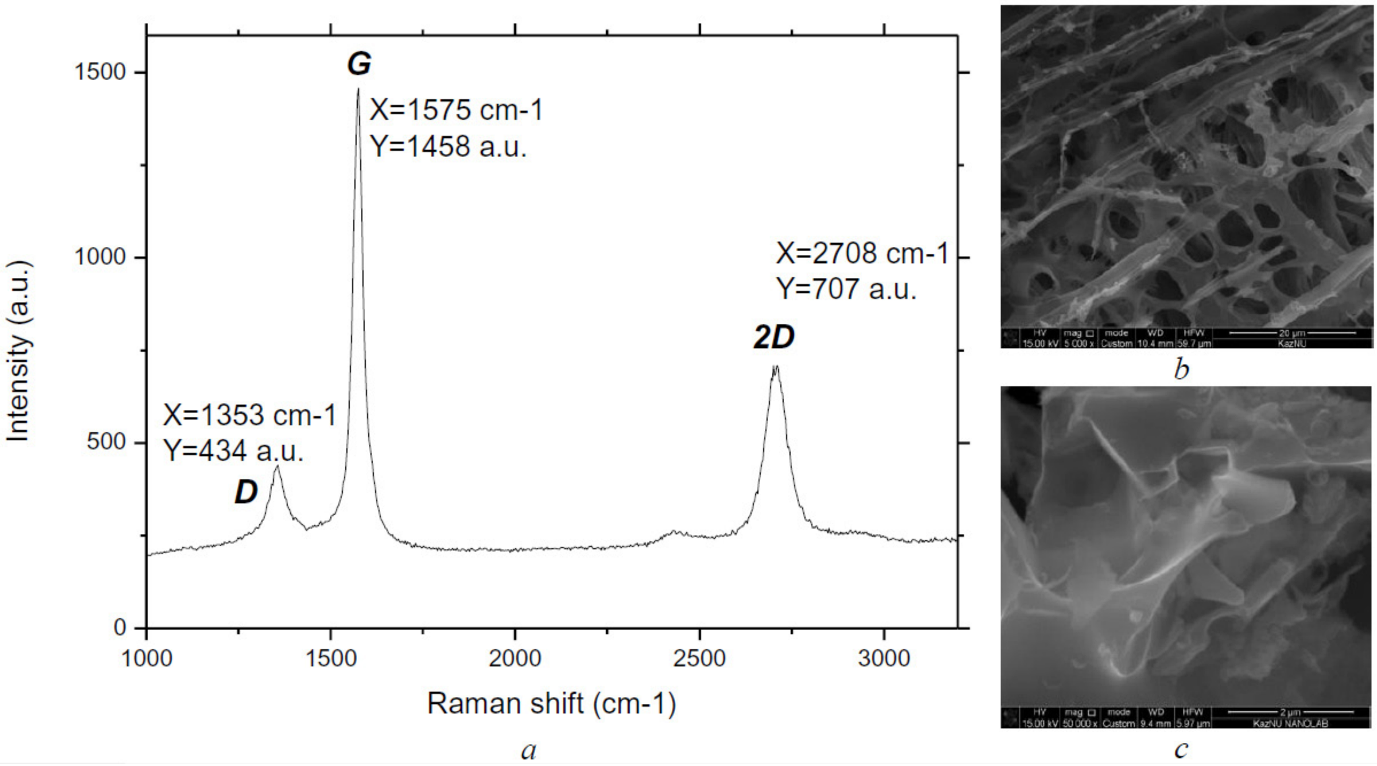

Figure 1 (a) Raman spectrum of a multilayer graphene and SEM microimages of the graphene film on areas of (b) 60 μm×50 μm and (c) 6 μm×5 μm.

As shown in Fig. 1b,c, graphene layers have a developed surface and this method generally does not affect the macrostructure of the samples, which is determined by the original structure of the RH. However, carbonization and chemical activation increase the specific surface of samples, as shown in previous studies [14].

IV. CONCLUSIONS

The method obtain graphene layers from rice husk was developed. Graphene layers were obtained from rice husk by potassium hydroxide activation followed by alkaline desilication. Rice husk samples were subjected to carbonization at the following conditions: 2 h of activation time, 850ºC and ratio of rice husk/KOH (wt/wt) was 1/4. Concentration of NaOH desilication solution was 1M. The obtained samples were studied using Raman spectroscopy; the peaks characterize the presence of graphene multilayers in the sample. The yield of the product was ~ 3% by weight.

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