



Effect of Surfactant Mass on the Production of Few-Layer Graphene from Nipah Shell Charcoal (FLG-Nyp) Using the Liquid Phase Exfoliation Method

Ilma Fadlilah^{1,*}, Ayu Pramita¹, Zhilal Shadiq¹

¹ Program Studi Teknologi Rekayasa Kimia Industri, Politeknik Negeri Cilacap, Kabupaten Cilacap, Indonesia

* Corresponding author: ilma.fadlilah@pnc.ac.id

<https://doi.org/10.14710/jksa.29.8.554-558>

Article Info

Article history:

Received: 09th December 2025

Revised: 07th September 2026

Accepted: 17th September 2026

Online: 30th September 2026

Keywords:

few layer graphene; LPE; Nipah;
linear alkylbenzene sulfonate

Abstract

This study aims to investigate the effect of surfactant mass on graphene production from Nipah shell charcoal (FLG-Nyp) via the Liquid Phase Exfoliation (LPE) method. The mass of Linear Alkylbenzene Sulfonate (LAS) surfactant was varied at 2 g, 2.5 g, and 3 g, with a fixed charcoal mass of 3 g and 250 mL of deionized water. The sonication process was carried out for three hours, after which the resulting suspension was dried at 105°C for two hours to obtain FLG powder. Characterization was performed using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and UV-Vis spectrophotometry. SEM results showed that adding up to 3 g of surfactant produced thinner FLG sheets with more homogeneous dispersion. XRD analysis revealed that the diffraction peak of FLG-Nyp₃ appeared at $2\theta = 26.50^\circ$, consistent with literature values, indicating the formation of an amorphous structure with the highest degree of exfoliation among the three variants. UV-Vis analysis exhibited a characteristic absorption band at 232 nm for all three samples. The results indicate that the exfoliation of graphitic carbon sheets increased with increasing surfactant mass.

1. Introduction

Graphene has become one of the most important topics in nanomaterials research due to its two-dimensional (2D) structure and unique combination of physical properties [1]. Graphene is generally classified as a few-layer structure, similar to single-walled, double-walled, or multi-walled carbon nanotubes. This is due to its highly flexible carbon framework, which facilitates functionalization and integration into various fields of application [2]. Graphene can be applied in a wide range of fields, including adsorbents, photovoltaic and photocatalytic applications of pollutants, and fluorescent sensors designed for the detection of pollutants, and electrochemical sensors [2, 3].

One source of charcoal for graphene production is biomass-derived charcoal. Nipah shell charcoal offers added value from a sustainability perspective. Nipah (*Nypa fruticans* Wurmb.) shells have a high carbon content and a lignocellulosic structure that can be converted into

activated charcoal or amorphous carbon material through carbonization [4, 5].

Various synthesis methods are used to produce graphene, including chemical vapor deposition, epitaxial growth on an insulator (SiC), reduction of graphene oxide, electrochemical methods, and sonochemical methods via liquid phase exfoliation (LPE) [6]. Among these methods, sonochemical via LPE is now widely used to produce new materials with unique characteristics. These superior properties are achieved because sonochemistry can produce particles that are significantly smaller and have a higher surface area than those produced by other techniques. Specifically, the sonochemical method works by directly exfoliating natural graphite using prolonged, mild sonication to produce defect-free multilayer graphene.

LPE is a simple, cost-effective method that can use environmentally friendly solvents [7]. LPE utilizes ultrasonic waves, in the presence of a surfactant, to break graphite layers by weakening van der Waals interactions

[8]. Surfactant-assisted LPE of graphite to produce dispersed graphene is one of the simplest, non-toxic, and environmentally friendly methods [9]. However, the efficiency of graphite exfoliation into graphene is strongly influenced by the presence of surfactant, which reduces interfacial tension and facilitates the separation of graphite layers [10].

The exfoliation of graphene layers can be facilitated by using the surfactant linear alkylbenzene sulfonate (LAS), which is found in commercial detergents. This surfactant weakens the interlayer Van der Waals forces in graphite, allowing the vibrations from the sonication process to effectively separate the graphene sheets. The use of this economical commercial detergent, rich in LAS surfactant, also serves as a solution to reduce the production costs of graphene [7].

Several previous studies have shown that the type and concentration of surfactant affect the yield and morphology of the graphene produced [10, 11]. Surfactants can modulate the surface energy of graphene, thereby enabling stable dispersion. While preventing graphene agglomeration, the volume effect of surfactants can also result in large distances between nearby graphene sheets, which affects the conductivity of graphene and the permeability of graphene membranes in the dispersion system [12].

Gayatri and Tjahjono [13] synthesized graphene from broiler chicken bone waste using the LPE method by varying the carbonization temperature (400°C and 600°C) and surfactant volume (0.76, 0.96, and 1.16 mL). Based on the UV-Vis characterization results, the greater the volume of surfactant added, the higher the absorbance. The UV-Vis spectra revealed that the best graphene material was obtained at a carbonization temperature of 400°C and a surfactant volume of 1.16 mL. However, studies on the effect of surfactant mass on the production of biomass-based graphene remain limited. Therefore, this work focuses on the effect of variations in the mass of the surfactant LAS, with the composition of graphite, surfactant, and deionized water, based on previous research [13, 14].

2. Experimental

2.1. Materials and Equipment

The main material used was Nipah shell charcoal previously carbonized at 300°C for 2 h, as reported in an earlier study [5]. Additional materials included linear alkylbenzene sulfonate (LAS) surfactant and deionized (DI) water. The equipment used consisted of a Taffware bath ultrasonic cleaner 60 W–40 KHz, filter paper 20–25 µm, oven, standard glassware, porcelain crucibles, UV-Vis-NIR spectrophotometer, X-ray Diffractometer (XRD), and Scanning Electron Microscope (SEM).

2.2. Synthesis Procedure for Graphene from Nipah Shell Charcoal (FLG-Nyp)

A total of 3 g of Nipah shell charcoal was mixed with LAS surfactant at 2 g (FLG-Nyp₂), 2.5 g (FLG-Nyp_{2.5}), and 3 g (FLG-Nyp₃), and 250 mL of deionized water [15]. The

mixture was sonicated for 3 h at room temperature to promote exfoliation. The sonicated suspension was then filtered and dried in an oven at 105°C for 2 h to obtain dry FLG-Nyp powder.

2.3. Characterization

The FLG-Nyp products were characterized using SEM to observe surface morphology, XRD to determine crystallinity, and UV-Vis-NIR spectrophotometry to evaluate the optical properties of FLG-Nyp.

3. Results and Discussion

Graphene was synthesized from Nipah shell charcoal using an ultrasonic cleaner with LAS surfactant to promote graphite exfoliation and produce thinner layers. Ultrasonic waves generate cavitation bubbles that create high local pressures, causing graphite layers to peel off and form graphene sheets. LAS surfactant must be added to the FLG-Nyp synthesis because the interlayer pressure between graphite layers is relatively low and exfoliation is difficult; therefore, a surfactant is required to assist the exfoliation process [16]. Based on the synthesis results, there was no significant change in the appearance of the suspension before and after sonication (Figure 1). As shown in the figure, the color of the suspension remained intensely black before and after sonication. The absence of a visible color change in graphene suspensions often indicates that the exfoliated material may have a low concentration [17].

3.1. Surface Morphology of FLG-Nyp by SEM

The surface morphology of FLG-Nyp, synthesized from Nipah shell charcoal with varying surfactant masses, is depicted in Figure 2. For FLG-Nyp₂, the surface structure appears partially open, with several thin layers, but stacking and irregularities remain visible, indicating that exfoliation is not yet fully uniform. In FLG-Nyp_{2.5}, the morphology shows more clearly defined, better-dispersed thin layers with minimal agglomeration. In FLG-Nyp₃, the morphology exhibits T-sheet-like structures with wrinkled edges, suggesting the formation of thinner layers. This observation is consistent with Chia *et al.* [18], who reported that graphene typically displays transparent or thin-layered structures. Based on these results, increasing the LAS surfactant mass results in thinner surface layers in FLG-Nyp.

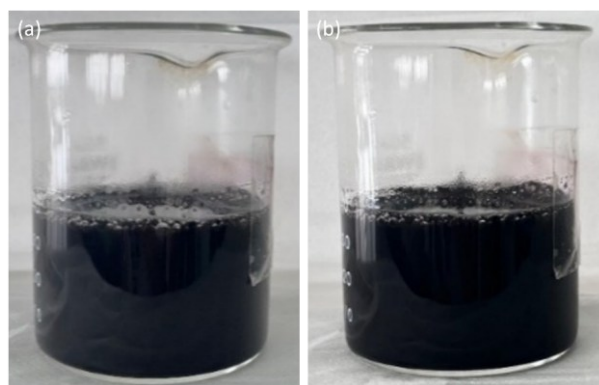


Figure 1. (a) before sonication (b) after sonication

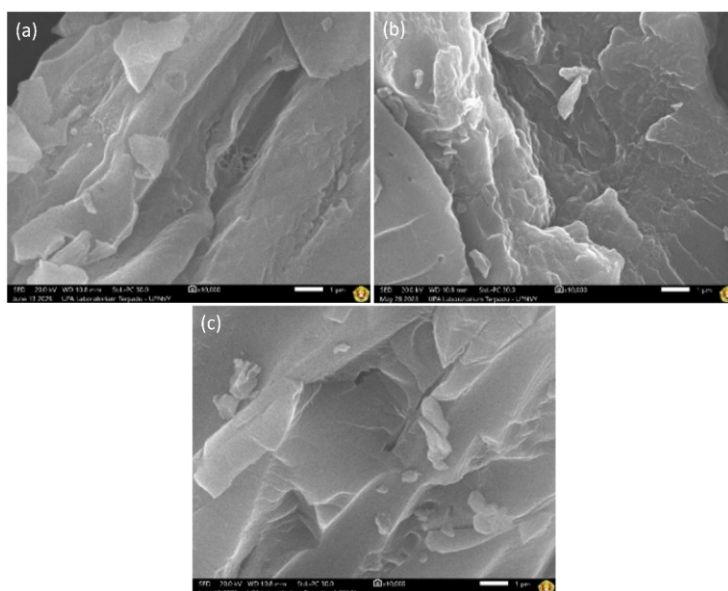


Figure 2. SEM micrographs at 10,000× magnification of (a) FLG-Nyp₂, (b) FLG-Nyp_{2.5}, and (c) FLG-Nyp₃

3.2. Crystallinity of FLG-Nyp by XRD

The structure of the graphene sheets obtained by ultrasonic exfoliation with an LAS surfactant was analyzed using XRD. As shown in Figure 3, all FLG-Nyp samples display broad diffraction peaks, indicating low crystallinity and the formation of amorphous structures. The XRD pattern of exfoliated graphene FLG-Nyp₂ shows a strong peak at $2\theta = 23.01^\circ$, corresponding to an interlayer distance (d) of 3.86 Å. FLG-Nyp_{2.5} exhibits a peak at $2\theta = 23.50^\circ$ with $d = 3.78$ Å, and FLG-Nyp₃ at $2\theta = 26.50^\circ$ with $d = 3.36$ Å. These results agree with Honorisal *et al.* [19], who reported that the diffraction peak of graphene occurs at $2\theta = 20\text{--}26^\circ$; these values and positions are characteristic of few-layer graphene (002) reflection. Another study on graphene synthesized using two different solvents reported diffraction peaks at $2\theta = 26.62^\circ$ and 26.22° [20].

Previous research that exfoliated graphite into graphene with the aid of an anionic surfactant found the graphene diffraction peak to be located at $2\theta = 22.1^\circ$ [21]. The decrease in interplanar spacing, as observed in FLG-Nyp_{2.5} and FLG-Nyp₃, may be attributed to the loss of oxygen functional groups and water molecules within the carbon layer structure [22]. The addition of oxygen functional groups such as hydroxyl, epoxide, and carboxyl, together with the presence of water molecules within the carbon structure, can increase the interlayer spacing (d -spacing). Conversely, the reduction of these functional groups, along with the loss of water molecules within the carbon structure, can decrease the interlayer spacing, thereby causing a shift in the interplanar spacing of graphene [23].

Based on the diffraction peaks in this study, increasing the mass of LAS surfactant in the synthesis of FLG-Nyp yielded progressively smaller d -spacing values. This indicates that the higher the mass of surfactant added, the purer the resulting FLG-Nyp material. This is evident from the diffraction peak of FLG-Nyp₃, which was obtained at the largest surfactant mass variation, namely 3 g, in range 2, consistent with the literature [19, 24].

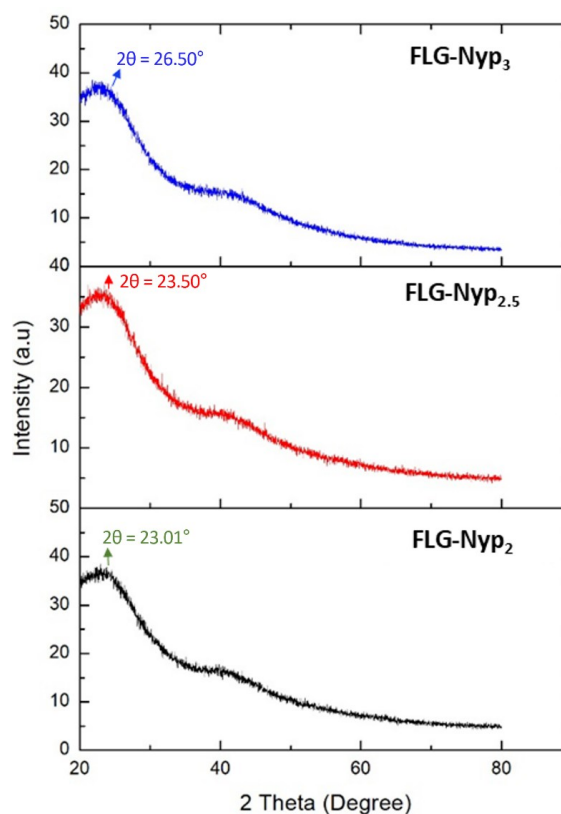


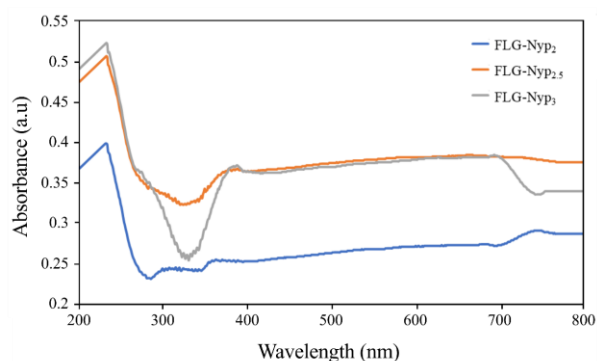
Figure 3. XRD diffractogram of FLG-Nyp samples

3.3. Optical Properties of FLG-Nyp by UV-Vis-NIR Spectrophotometer

The optical properties of the synthesized FLG-Nyp were analyzed using a UV-Vis-NIR spectrophotometer. The analysis of solid samples for maximum wavelength determination was performed using a UV-Vis-NIR spectrophotometer via DRS. The solid FLG-Nyp samples were packed into the sample holder until compact and level at the surface, and placed at the measurement port of the integrating sphere. The wavelength range used was 200–800 nm, encompassing the ultraviolet and visible regions. The measurement results for FLG-Nyp are presented in Table 1 and Figure 4.

Table 1. Absorbance and $\lambda_{\max}$ of FLG-Nyp

Materials	Absorbance	$\lambda_{\max}$ (nm)
FLG-Nyp ₂	0.400	232
FLG-Nyp _{2.5}	0.508	232
FLG-Nyp ₃	0.524	232

**Figure 4.** UV-Vis spectra of FLG-Nyp samples

UV-Vis absorption analysis provides information on conjugation and bonding in graphene. Based on Table 1 and Figure 4, identical absorption peaks at 232 nm are observed for all three FLG-Nyp samples. The absorption peaks of FLG-Nyp in the 200–300 nm range correspond to the $\pi-\pi^*$ absorption band. The FLG-Nyp absorption peak around ~230 nm is associated with $\pi-\pi^*$ transitions occurring during graphite exfoliation [18]. The $\pi-\pi^*$ transitions can arise from chromophore groups associated with C=C bonds in graphene. The FLG-Nyp absorbance peak at ~232 nm is characteristic of graphene, which typically appears in the range of 220–270 nm [14, 25].

4. Conclusion

This study demonstrates that the mass of the LAS surfactant influences the exfoliation of graphite into FLG-Nyp. Increasing the surfactant mass resulted in progressively thinner FLG-Nyp layers, with FLG-Nyp₃ exhibiting the most pronounced exfoliation among the investigated formulations. These findings indicate that a LAS surfactant mass of 3 g was the most effective condition for graphite exfoliation under the experimental conditions employed. The results provide a basis for controlling graphite exfoliation via surfactant-assisted processing in the preparation of FLG-Nyp.

Acknowledgment

This research was funded by the Directorate of Research and Community Service – Ministry of Higher Education, Science, and Technology through the 2025 Higher Education Operational Assistance Research Grant, Contract Number 169/C3/DT.05.00/PL-BATCH II/2025.

References

- [1] A. Jiříčková, O. Jankovský, Z. Sofer, D. Sedmidubský, Synthesis and Applications of Graphene Oxide, *Materials*, 15, 3, (2022), 920 <https://doi.org/10.3390/ma15030920>
- [2] Faiza Asghar, Bushra Shakoor, Saira Fatima, Shamsa Munir, Humaira Razzaq, Shazia Naheed, Ian S. Butler, Fabrication and prospective applications of

graphene oxide-modified nanocomposites for wastewater remediation, *RSC Advances*, 12, 19, (2022), 11750–11768 <https://doi.org/10.1039/d2ra00271j>

- [3] Meijiao Lü, Jing Li, XuYu Yang, ChangAn Zhang, Jia Yang, Hao Hu, XianBao Wang, Applications of graphene-based materials in environmental protection and detection, *Chinese Science Bulletin*, 58, (2013), 2698–2710 <https://doi.org/10.1007/s11434-013-5887-y>
- [4] Susinggih Wijana, Hendrix Yulis Setyawan, Zhijian Wan, Mingming Zhu, Dodyk Pranowo, Ika Atsari Dewi, Mega Permata Nareswari, The potential of Nypa Frutican as an energy source in Indonesia: A review, *Advanced Food Science, Sustainable Agriculture and Agroindustrial Engineering*, 6, 1, (2023), 88–96 <http://doi.org/10.21776/ub.afssaae.2023.006.01.8>
- [5] I. Fadlilah, N. A. Triwuri, A. Pramita, Perbandingan Karbon Aktif-Tempurung Nipah dan Karbon Aktif-Kulit Pisang Kepok Teraktivasi Kalium Hidroksida, *CHEESA: Chemical Engineering Research Articles*, 5, 1, (2022), 20–27 <https://doi.org/10.25273/cheesa.v5i1.10992.20-27>
- [6] L. J. Ling, C. S. Yee, M. Jaafar, Effect of sonication time on the properties of multilayer graphene, *AIP Conference Proceedings*, 2017 <https://doi.org/10.1063/1.4993323>
- [7] A. A. Fikri, A. N. Aisyah, S. Alfarisa, W. S. B. Dwandaru, Synthesis of Graphene Oxide via Liquid Exfoliation Using Self-Custom-Made Tweeter Piezoelectric Ultrasound Generator and Assisted by Surfactant from Commercial Detergent, *American Journal of Applied Sciences*, 13, 11, (2016), 1129–1135 <https://doi.org/10.3844/ajassp.2016.1129.1135>
- [8] P. Chavalekvirat, W. Hirunpinyopas, K. Deshsorn, K. Jitapunkul, P. Iamprasertkun, Liquid Phase Exfoliation of 2D Materials and Its Electrochemical Applications in the Data-Driven Future, *Precision Chemistry*, 2, 7, (2024), 300–329 <https://doi.org/10.1021/prechem.3c00119>
- [9] F. S. Ismail, K. Yusoh, A. S. Zainal Abidin, A. H. Abdullah, Z. Ismail, Three different types of tea as surfactant in liquid exfoliation of graphite: Green tea, black tea and oolong tea, *IOP Conference Series: Materials Science and Engineering*, 2019 <https://doi.org/10.1088/1757-899X/702/1/012036>
- [10] K. C. B. Maia, A. Densy dos Santos Francisco, M. P. Moreira, R. S. V. Nascimento, D. Grasseschi, Advancements in Surfactant Carriers for Enhanced Oil Recovery: Mechanisms, Challenges, and Opportunities, *ACS Omega*, 9, 35, (2024), 36874–36903 <https://doi.org/10.1021/acsomega.4c04058>
- [11] R. Narayan, S. O. Kim, Surfactant mediated liquid phase exfoliation of graphene, *Nano Convergence*, 2, (2015), 20 <https://doi.org/10.1186/s40580-015-0050-x>
- [12] Liangchuan Li, Ming Zhou, Long Jin, Lincong Liu, Youtang Mo, Xiao Li, Zhaoyou Mo, Zhenzhao Liu, Shengli You, Hongwei Zhu, Research Progress of the Liquid Phase Exfoliation and Stable Dispersion Mechanism and Method of Graphene, *Frontiers in Materials*, 6, (2019), 325 <https://doi.org/10.3389/fmats.2019.00325>
- [13] A. S. Gayatri, A. Tjahjono, Pengaruh temperatur pembakaran dan volume surfaktan pada proses

- sintesis graphene oxide (GO) dari limbah tulang ayam broiler, *Jurnal Teras Fisika*, 5, 1, (2022), 260-266 <https://doi.org/10.20884/1.jtf.2022.5.1.5497>
- [14] R. Santika, A. Astuti, S. R. A. Usna, Sintesis dan Karakterisasi Sifat Optik Graphene Oxide dari Limbah Bulu Ayam dengan Metode *Liquid Phase Exfoliation*, *Jurnal Fisika Unand*, 12, 2, (2023), 228-234
- [15] Zahra Izzati Aziz, Arif Tjahjono, Sintesis Graphene Oxide (GO) berbasis arang dengan metode *liquid phase exfoliation* (LPE), *Jurnal Teras Fisika*, 5, 2, (2022), 1-8
<https://doi.org/10.20884/1.jtf.2022.5.2.5488>
- [16] Xiaoguang Gu, Yue Zhao, Kai Sun, Carolina L. Z. Vieira, Zhijuan Jia, Chi Cui, Zhenjun Wang, Annika Walsh, Shaodan Huang, Method of ultrasound-assisted liquid-phase exfoliation to prepare graphene, *Ultrasonics Sonochemistry*, 58, (2019), 104630
<https://doi.org/10.1016/j.ultsonch.2019.104630>
- [17] Huailiang Xu, Zhikai Qi, Hongchang Jin, Jinxi Wang, Yan Qu, Yanwu Zhu, Hengxing Ji, Identification of graphene oxide and its structural features in solvents by optical microscopy, *RSC Advances*, 9, 32, (2019), 18559-18564
<http://doi.org/10.1039/c9ra02076d>
- [18] Joanna Su Yuin Chia, Michelle T. T. Tan, Poi Sim Khiew, Jit Kai Chin, Hingwah Lee, D. C. S. Bien, Chiu Wee Siong, A novel one step synthesis of graphene via sonochemical-assisted solvent exfoliation approach for electrochemical sensing application, *Chemical Engineering Journal*, 249, (2014), 270-278
<https://doi.org/10.1016/j.cej.2014.03.081>
- [19] M. B. P. Honorisal, N. Huda, T. Partuti, A. Sholehah, Sintesis dan karakterisasi grafena oksida dari tempurung kelapa dengan metode sonikasi dan hidrotermal, *TEKNIKA: Jurnal Sains dan Teknologi*, 16, 1, (2020), 1-11
<https://doi.org/10.36055/tjst.v16i1.7519>
- [20] Al-Sayed Al-Sherbini, Mona Bakr, Iman Ghoneim, Mohamed Saad, Exfoliation of graphene sheets via high energy wet milling of graphite in 2-ethylhexanol and kerosene, *Journal of Advanced Research*, 8, 3, (2017), 209-215
<https://doi.org/10.1016/j.jare.2017.01.004>
- [21] Kang Zhang, Xiaohua Zhang, Hengxiang Li, Xiaohan Xing, Li'e Jin, Qing Cao, Ping Li, Direct exfoliation of graphite into graphene in aqueous solution using a novel surfactant obtained from used engine oil, *Journal of Materials Science*, 53, (2018), 2484-2496
<http://doi.org/10.1007/s10853-017-1729-7>
- [22] C. Simanjuntak, R. Siburian, H. Marpaung, Tamrin, Properties of Mg/graphite and Mg/graphene as cathode electrode on primary cell battery, *Heliyon*, 6, 1, (2020), e03118
<https://doi.org/10.1016/j.heliyon.2019.e03118>
- [23] R. Siburian, H. Sihotang, S. L. Raja, M. Supeno, C. Simanjuntak, New Route to Synthesize of Graphene Nano Sheets, *Oriental Journal of Chemistry*, 34, 1, (2018), 182-187
<http://dx.doi.org/10.13005/ojc/340120>
- [24] Václav Štengl, Preparation of Graphene by Using an Intense Cavitation Field in a Pressurized Ultrasonic Reactor, *Chemistry – A European Journal*, 18, (2012), 14047-14054
<https://doi.org/10.1002/chem.201201411>
- [25] Yuditha Ignasia Bete, Minsyahril Bukit, Albert Zicko Johannes, Redi K. Pingak, Kajian Awal Sifat Optik Graphene Oxide Berbahan Dasar Arang Tongkol Jagung yang Disintesis dengan Metode *Liquid Phase Exfoliation* (LPE), *Jurnal Fisika: Fisika Sains dan Aplikasinya*, 4, 2, (2019), 114-120
<https://doi.org/10.35508/fisa.v4i2.1832>