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Synthesis and characterisation of oil palm fibre-based graphene deposited on copper particles for superlubricity oil additive

Nurul Liyana Rahman [1], Hilmi Amiruddin [1,2*], Mohd Fadzli Bin Abdollah [1,2], Noritsugu Umehara [3]

[1] Fakulti Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, MALAYSIA.
[2] Centre for Advanced Research on Energy, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, MALAYSIA.
[3] Department of Micro-Nano Mechanical Science and Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, JAPAN.
[*] Corresponding author: hilmi@utem.edu.my

KEYWORD

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ABSTRACT

This paper presents the synthesis and characterisation of oil palm fibre-based graphene deposited on copper (Cu) particles for superlubricity oil additive. Oil palm fibre (OPF) is employed as a renewable carbon feedstock to grow graphene on Cu particles during Chemical Vapor Deposition (CVD) process. The surface morphology was observed using Scanning Electron Microscopy (SEM) embedded with Energy Dispersive X-Ray Analysis (EDX), and Transmission Electron Microscopy (TEM). Meanwhile, the presence and quality of graphene were validated and examined using Gaussian analysis of Raman spectroscopy. The results show that decreasing the peak D to peak G intensity ratio (I_D/I_G) tends to improve graphene quality with less number density of structural defects. The I_{2D}/I_G value greater than 1 is indicative of multilayer graphene growth. Increasing the full width at half maximum (FWHM) ratio of peak D to peak G ($FWHM_D/FWHM_G$) suggests a better dispersion of graphene. The uniform spread of surface 'wrinkles' observed by SEM and TEM implies that the distribution of graphene is more homogeneous on the surface of Cu particles. Further investigation into the synergistic effect of graphene as an oil additive can be performed to achieve superlubricity properties.

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1.0 INTRODUCTION

It has recently been demonstrated that graphene has extraordinary mechanical properties such as high strength, hardness, fracture toughness, and non-dangerousness. Because of its exceptional nanotribological characteristics, graphene has recently become a popular lubricant additive (Paul et al., 2019). A large number of studies on the effects of nanoparticles as oil additives have been published. Many studies show that adding nanoparticles can reduce friction and wear even at concentrations as low as 1.0wt%. According to (Uflyand et al., 2019), many nanomaterials utilised as lubricating oil additives have been developed, mainly carbon, carbon nanotubes, graphene oxide, boron nitrides, and silicone oxide. However, the most extensive research has been conducted using metal-containing nanomaterials and nanoparticles, such as metal, its oxides, and sulphides. Copper (Cu) nanoparticles, an eco-friendly additive, garnered special attention due to their excellent lubricity performance (Borda et al., 2018).

Although graphene has recently received a lot of interest for its lubricating qualities, graphene liquid superlubricity, where the coefficient of friction (COF) is less than 0.01 can only be attained with water-based lubricants (Liu et al., 2020). Superlubricity properties in the context of the tribological behaviour of graphene additives in oil remain a significant challenge. This is because lubricating oil has a higher viscosity than water-based lubricants, and the graphene additive, which is typically in flakes form, is harder to disseminate and stabilise in oil, making its superlubricity capabilities less efficient. Thus, the synthesis of sustainable and high-quality multilayer graphene on nanoparticle surfaces with low defect density offers a synergistic effect on superlubricity, which can be exploited as an oil additive in future study (Baheshti et al., 2020).

Aside from that, graphene is unique due to its sp^2 hybridisation and incredibly thin atomic thickness, which gives it good mechanical strength and hardness. Graphene is an isolated single layer of carbon consisting of sp^2 hybridized C-C bonding with π-electron clouds. The bond in between carbon-to-carbon atoms arranged in a hexagonal ring structure. The layers are bonded together by weak van der Waals forces, thus allowing it to disperse well. From the engineering point of view, thin flakes composed of few layers of carbon atoms, including mono-layer graphene, can be very essential due to their intriguing structure and physical properties, as well as promising future applications in technical domains.

Since the first successful separation of monolayer graphene from graphite, graphene has drawn much attention from researchers in various kinds of fields (Novoselov et al., 2004). Single-layer graphene is a potential material due to its chemical stability and high mechanical strength makes it versatile in many applications. However, the importance of multilayer graphene has also rapidly grown for the past few years because of its unique functionality and wide applicability in various fields, for example in electrical nanodevices. Besides, multilayer graphene has lower friction than monolayer graphene, according to experiments and simulations (Li et al., 2016). It is also claimed that multilayer graphene can improve tribological properties by accelerating the production of lubricating film adsorbed on the wear surface during the friction process (Liu et al., 2020).

Researchers explored a variety of methods for graphene synthesis to improve its mechanical and tribological properties (Venturi et al., 2019; Kumar et al., 2017; Elias et al., 2009). Chemical Vapor Deposition (CVD) is a widely used process for synthesising graphene (Li et al., 2009). Furthermore, CVD is a technology that induces the deposition of a thin film through a chemical reaction. The CVD process is the most cost-effective and simple way to produce high-quality multilayer graphene. Cu, a transition metal, will serve as both a catalyst and a substrate for graphene growth in the CVD process. Cu is widely used as a substrate due to the limited solubility of carbon and copper at high temperatures (Shi et al., 2020). In the CVD process, inert gases like as nitrogen and argon are typically used to transport carbon vapour to the substrate surface. However, hydrogen is employed because it contributes significantly to the growth of high-quality graphene. Growth temperature and hydrogen flow rates can be employed to improve the homogeneity and defect density of graphene (Saeed et al., 2020).

Other than that, graphene of various qualities also can be grown from a variety of carbon-containing feedstocks, an economically feasible and compatible method which may be of great significance for industrial applications. In this regard, from technological and environmental aspects, it may be of great significance to use agricultural wastes such as oil palm fibre (OPF) as a carbon feedstock to synthesise high-quality graphene, thereby converting waste into high value-added products. From a previous study (Mat Tahir et al., 2020), CVD synthesised graphene on a Cu plate substrate using an oil palm fibre feedstock shows a lower COF about 0.03 and wear (≈1.0 x 10^{-8} mm^3/Nm) under dry sliding conditions than other synthesised graphene from different feedstocks. OPF is a promising carbon precursor due to its abundance, low cost, and high carbon content. It contains lignin and cellulose, which are the main components that can be converted to carbon.

As discussed above, this study aims to synthesise and characterise the graphene growth from OPF feedstock deposited on copper particles surfaces for use as a superlubricity oil additive.

2.0 EXPERIMENTAL PROCEDURE

Graphite (G) particles with average of 20 μm utilised as removal spacers were consistently mixed with Cu particles (average of 20 μm) using a speed mixer to prevent Cu particle coalescence at high temperatures for graphene growth. The average size of syntesised graphene particle is 700 μm. The particle size distribution, as shown in Figure 1, of synthesised graphene is bigger than copper and graphite because of the aggregation which refers to the process of particles sticking together due to various forces such as Van der Waals forces, electrostatic attraction, or intermolecular bonding.

Prior to the synthesis process, the Cu particle was washed with acetone in an ultrasonic bath for 5 minutes and left dried. The purpose of this step is to ensure there is no oxide layer on Cu particles and remove the impurities on Cu particles surfaces. Moreover, hydrogen that is used in synthesis process was significantly important in removing the oxide layer on the metal surface. Then, the synthesis was carried out using the CVD method and the schematic diagram illustrated in Figure 2.

A mixture weight percent ratio of G:Cu (6:4) was placed into a CVD chamber at Furnace 2, which was annealed to 1020°C for 30 minutes at a heating rate of 60°C/min with varied hydrogen (H_2) flow rates ranging from 400 to 800 cc/min. During the annealing step, H_2 is often used because it removes the oxide layer from the metal surface. Therefore, it is essential in the cleaning and crystallisation of metal substrates. Furthermore, molecular hydrogen is important in the graphene growth process. Accordingly, variable H_2 flow rates will have an effect on the quality of the grown graphene. After annealing process at Furnace 2, 20g of OPF feedstock was heated (Furnace 1) at 1000^{o}C to grow graphene with continuous H_2 flow under atmospheric pressure, and the system was then cooled at an 80°C/min cooling rate. The growth time is 30 minutes.

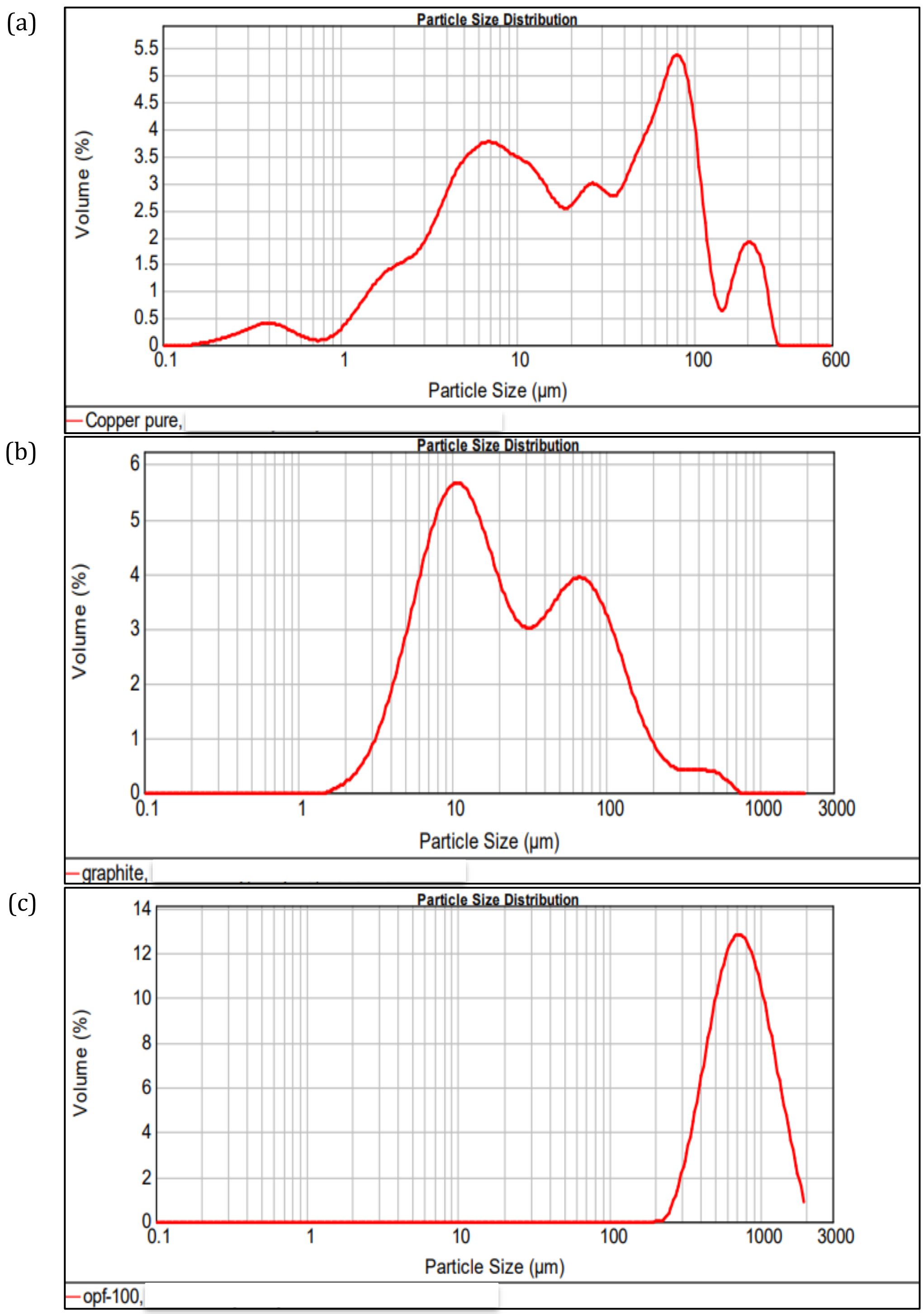

Figure 1: Particle size analysis of (a) copper, (b) graphite and (c) synthesised graphene particles.

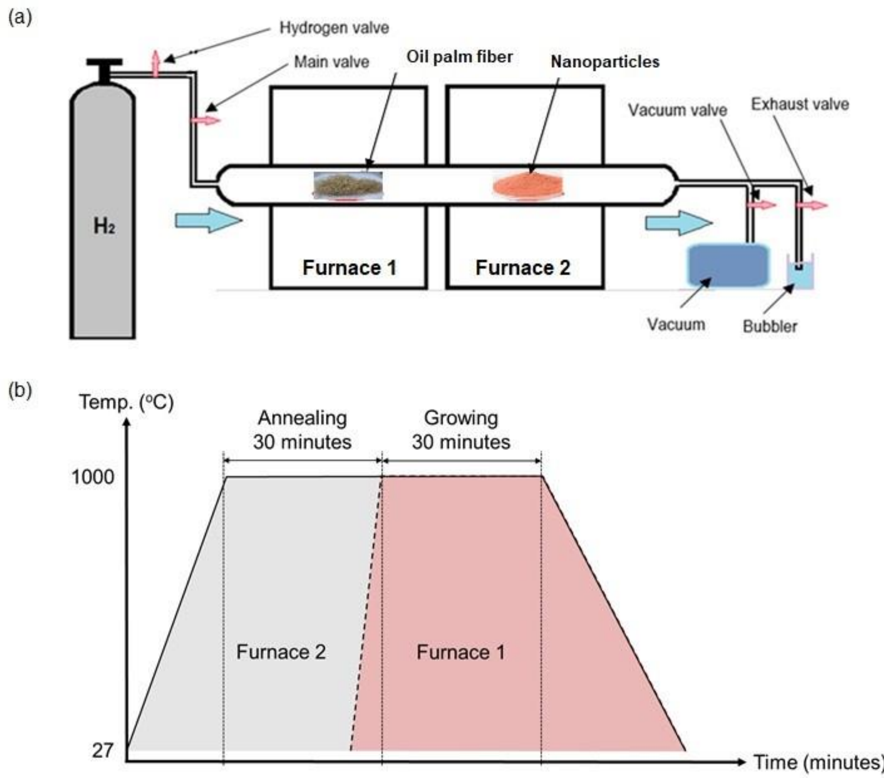

Figure 2: (a) Schematic diagram of the tube furnace for CVD process and (b) the deposition process on the Cu particles.

Theoretically, graphene on Cu is grown by the decomposition of solid carbon precursor in a dilute hydrogen atmosphere over the surface at 1000°C. Typically, during the growth process, the Cu substrate will be first annealed for 30 minutes at 1000°C in a dilute hydrogen environment. The carbon source precursor is then introduced at a modest flow rate for about 30 minutes.

Figure 3 illustrates the proposed growth mechanism of graphene on Cu substrate. The purpose of high temperature annealing in an H_2 environment is to remove the natural oxide layer on the Cu surface, while Cu grains form. When Cu substrate is exposed to a H_2 environment at 1000°C, graphene islands form at random but predominantly at the grain boundary of the Cu surface during nucleation process. As the graphene domains grow in size, they ultimately cover the entire area of the Cu substrates and aggregate into a continuous graphene film.

In the context of CVD graphene deposition, a metal substrate such as Cu is heated in a quartz furnace tube under a flow of hydrogen at low vacuum or atmospheric pressure. The used of transition metal substrates in CVD has been identified as the most promising, cost-effective, and feasible technique for producing single layer or multi-layer graphene. Rouff's group showed CVD graphene using Cu as a transition metal substrate for single layer graphene synthesis for the first time in 2009 (Li et al., 2009). Due to the low solubility of carbon in Cu, which results in a self-limited process, CVD using Cu as one of the fastest developing methods for producing single layer graphene (Li et al., 2009).

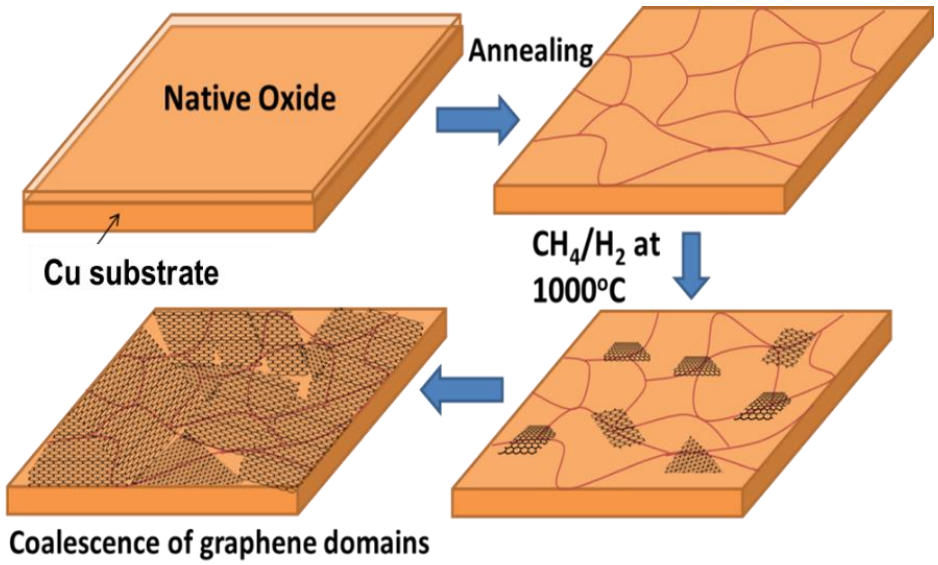

Figure 3: A schematic diagram of the growth mechanism of graphene on Cu substrates by CVD method

While pre-processing, annealing and longer growth times have been shown to improve graphene quality, a middle ground between quality and processing time is required to make CVD grown graphene economically feasible. Hydrogen is commonly used in the annealing process because it removes the oxide layer on the metal surface. As a result, it plays an important role in the cleaning and crystallisation of metallic surfaces. Yang et al., 2006 reported that annealing Cu under H_2 atmosphere increased the nucleation density and consequently promoted multilayer graphene domains located mostly along the rolling lines. Molecular hydrogen (H_2) is also essential in the graphene growth process. The higher the hydrogen flow rate, the faster the growth of graphene. This is because increasing the H_2 feed in the reactor increases the number of active sites on the surface of the Cu substrate. In a recent study by Ibrahim et al., different hydrogen flow rates were used in the annealing step, demonstrating that the surface morphology of the growth substrate (Cu in this case) in the absence of hydrogen exhibited step-like structure, whereas in the presence of hydrogen, the surface becomes smoother with some surface defects (Ibrahim et al., 2015).

The graphene deposited on the surfaces of Cu particles was then observed using Scanning Electron Microscopy (SEM) embedded with Energy Dispersive X-Ray (EDX) for surface morphology and chemical composition analysis. Transmission Electron Microscopy (TEM) has also been used to characterise surface morphology. Additionally, Raman spectroscopy analysis was used for quantitative analysis. Each sample was tested thrice by using Raman spectroscopy analysis for a comparison. The data were chosen by choosing the best spectrum of Raman spectra for further analysis.

3.0 RESULTS AND DISCUSSION

3.1 Surface Morphology

In graphene research, SEM is commonly used to characterize graphene grown on conductive substrates using the CVD method. As we know, it provides useful information about graphene growth rates, sample coverage, nucleation density, grain size, and morphology, but it cannot determine the exact number of graphene layers where can only provide an estimate of the uniformity of the graphene layers. However, the contrast in SEM images provide qualitative information about the thickness of the deposited graphene, where the darker areas are covered with a higher number of graphene layers while the lighter area are covered with fewer graphene layers (Saeed et al., 2020). As a result, regions of higher uniform contrast suggest better graphene film coverage with more homogeneous graphene thickness and layer numbers. Besides, the contrast of CVD graphene SEM pictures varies with the amount of graphene layers which low contrast suggests few graphene layers. This contrast is due to the quantity of secondary electrons generated in the upper few nanometers of the sample surface.

Figure 3 shows micrograph images of graphene synthesized on Cu particle surfaces at varying H_2 flow rates. The surfaces are observed to have ripples and wrinkles. This means that the graphene distribution on the surface of Cu particles is more homogeneous. The darker areas also can be easily distinguished on the surface of the copper substrate. The darker area represents two or more layers. The dark-coloured regions represent the multilayered graphene that served as the seed layer (Zhang et al., 2010). This also implies that the more layers of oriented carbon atoms on the surface of copper, the lower the electron yield and the darker the regions appear on the image. Further determination on the number of graphene layers can be identified from the position and shape of the 2D band in Raman spectroscopy analysis.

The accelerating voltage used for SEM is 10kV. Accelerating voltage is also important in graphene characterization using SEM, as the usual condition is 5kV. Higher accelerating voltages typically enhance lateral sharpness and signal-to-noise ratio while increasing interaction volume and signal penetration depth from the sample's surface. Graphene folding lines were brighter and more distinct than monolayer graphene, and bright spots were observed, which are thought to be defects in the graphene sheet that may produce more secondary electrons (Lee et al., 2015). As a result, single-layer graphene and multilayer graphene can be easily distinguished.

The carbon content on the particle surfaces increased dramatically with H_2 flow rates, according to the EDX results (Table 1). It also implies that, despite the increased carbon concentration, the oxygen content remains constant. The oxygen content is nearly zero as the hydrogen flow rates increase. Means that, as the hydrogen flow rates increase the composition of oxygen becomes smaller and indistinct. Thus, the oxygen was assumed to disappear at higher hydrogen flowrate and it remains constant as the hydrogen flow rates increase. This suggests that increasing carbon content on the substrate with increased H_2 flow rate improves graphene quality.

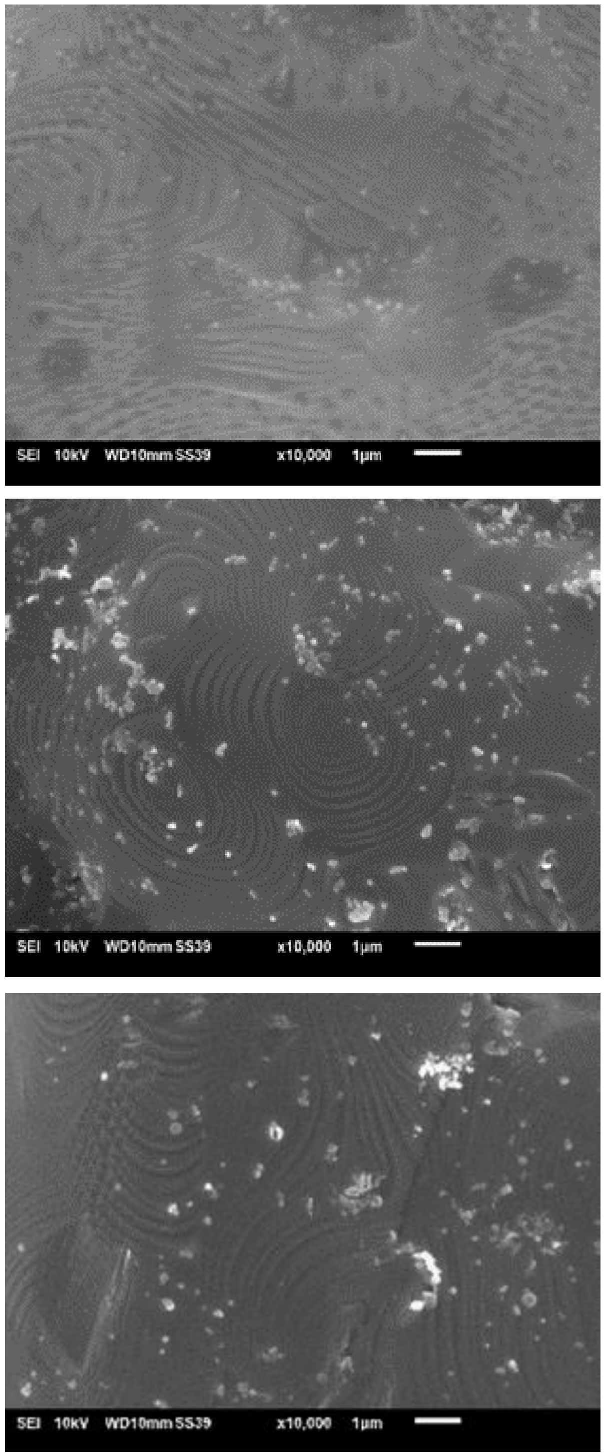

Figure 4: SEM images of synthesised graphene deposited on copper particles at H_2 flow rates of (a) 400 cc/min, (b) 600 cc/min, and (c) 800 cc/min.

Table 1: EDX results on element composition at various H_2 flow rates.

Sample	Flow rates of H_2 [cc/min]	Weight%		
		Carbon	Oxygen	Copper
A	400	71.29	6.34	22.27
B	600	87.10	0.00	12.90
C	800	94.74	0.00	5.26

TEM was subsequently used to characterise the surface morphology of synthesised graphene, as shown in Figure 4. The accelerating voltage used for graphene characterization in TEM is 200kV. From the Figure 4, a clear view of the images revealed that the samples had multiple layers because the substance was produced in powder form. Graphene is a transparent substance that is stable in the presence of high-energy electrons. The graphene image had a thin flat flake and crumpled shape, which was consistent with previous findings (Geng et al., 2009; Dideykin et al., 2011). The wrinkled flake structure, as well as the thin film morphology of graphene sheets, were clearly visible. The thin multilayers and crumpled sheets were formed by the rapid elimination of intercalated oxygen and other functional groups between the layers during the exfoliation process in the reduction and sonification medium (Mishra & Kamaprabhu, 2011). Therefore, the impacts of exfoliation on the disordered solid graphene structure were observed in the form of crumpled sheets.

3.2 Raman Spectroscopy Analysis

Raman spectroscopy is one of the techniques for the structural characterisation of the graphitic materials. The two unique Raman features in almost all crystalline sp2 materials are G-bands and 2D-bands which the latter also known as the G'-band. The number of dispersion events involved determines the order of a Raman band; for example, the G band is first order, whereas the 2D band (two phonons) and D band (one phonon and a defect) are second order. In comparison to monolayer graphene, the 2D peak intensity reduces as the number of layers grows, becoming broader and upshifted. Graphene has a unique single, a sharp 2D peak that is approximately four times as intense as the G peak. The D indicates the defective and impure structure of graphite sheets. The G band indicates the order and purity of graphite sheets. The 2D band is the characteristics peak of graphene structure.

Figure 6 shows a Gaussian analysis of Raman spectroscopy. The carbon produced at each flow rate is slightly variable because the D, G, and 2D band peaks are slightly slanted. Furthermore, the height of each of these peaks varies. Sharma et al., 2014 and Ji et al., 2011 discovered that graphene was anticipated to have a G band peak at around 1580 cm^{-1} and a 2D band peak at roughly 2730 cm^{-1}, but no distinguishing D band peak. Graphene has the highest peak at flow rates of 800 cc/min, followed by 400 cc/min and 600 cc/min. According to Saeed et al., 2020, growth temperature and hydrogen flow rates can regulate graphene quality in terms of uniformity and defect density. Molecular hydrogen is essential in the graphene growth process and has an impact on graphene quality. As a result, the higher the hydrogen flow rates, the better the quality of graphene grown.

(a)
(b)
(c)

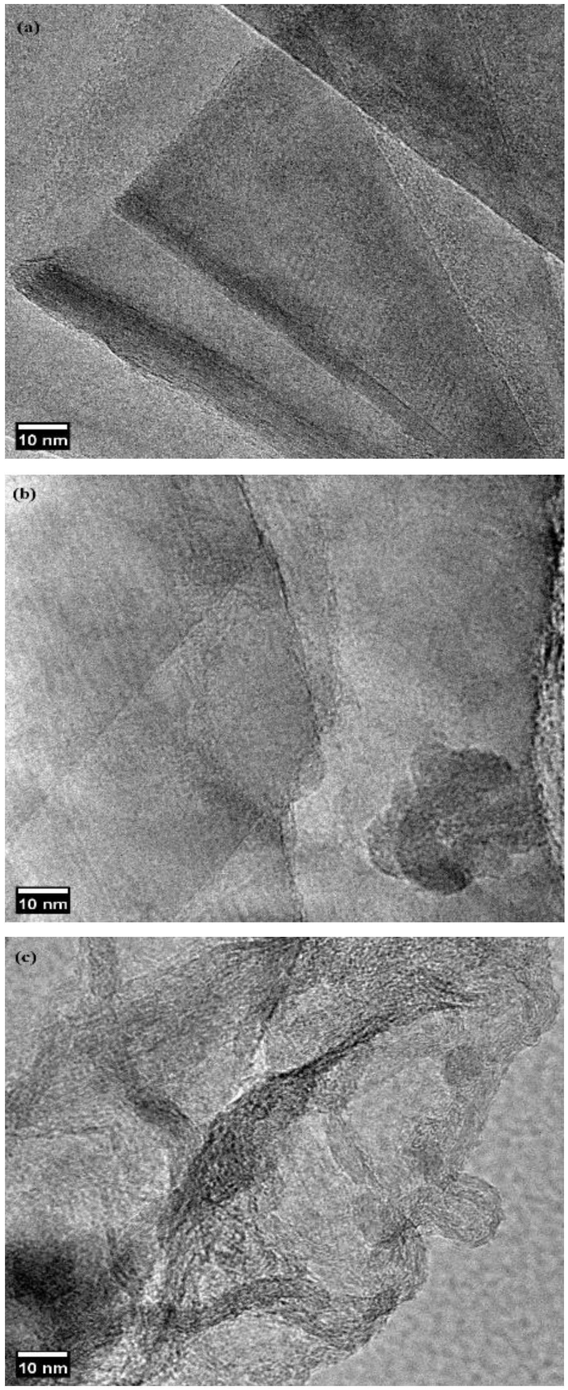

Figure 5: TEM images of synthesised graphene deposited on copper particles at H_2 flow rates of (a) 400 cc/min, (b) 600 cc/min, and (c) 800 cc/min.

Table 2 displays the quantitative values derived from Raman spectra by Gaussian analysis. According to the findings, the I_D/I_G decreases as the H_2 flow rate increases. This implies that the quality of synthesised graphene was improved by having a low density of structural defects. From Le et al., 2013 they found that for growing time of 30 min, the ratio I_D/I_G is lowest (with the value of 0.16) indicates that the quality of the graphene is the best. Thus, the flow rates of 800 cc/min was chosen as the best flow rates in producing the best quality of graphene. The intensity ratio of I_G to I_{2D} is known to be depending on the number of graphene layers (Reina et al., 2009; Dong et al., 2011). The ratio I_G/I_{2D} ~ 0.3-0.5 is for monolayer graphene, $0.5 < I_G/I_{2D} < 1$ for bilayer graphene, and $I_G/I_{2D} > 1$ for multilayer graphene. Based on the ratio I_G/I_{2D} of Raman spectra in the Table 2, it can be concluded that the graphene films grown on the Cu particles for all flow rates are multilayers. As for the number of layers in multilayer graphene were determine from Bianco et al., 2013. It is found that graphene can be categorized based on the number of graphene layer (L) which stacked as monolayer graphene (1 L), few-layer graphene (<5 L), multilayer graphene (<10 L) and graphene nanoplatelets (>10 L). Thus, the average number of layers found is between 5 – 10 layers.

Meanwhile, as the H_2 flow rate increased, so did the value of the full width at half maximum (FWHM) ratio of peak D to peak G ($FWHM_D/FWHM_G$). It was demonstrated that graphene dispersion had improved. Therefore, this study suggests that increasing the H_2 flow rate enhances the quality of synthesised graphene with the lowest defect density.

Table 2: The quantitative values derived from Raman spectra by Gaussian analysis.

Sample	**H_2 Flow rate**	**I_D**	**I_G**	**I_{2D}**	**I_D/I_G**	**I_G/ I_{2D}**	**$FWHM_D$**	**$FWHM_G$**	**$FWHM_D$/ $FWHM_G$**
A	400	354.42	2331.54	328.69	0.15	7.09	49.07	23.71	2.07
B	600	504.33	4867.67	717.54	0.10	6.78	49.19	23.16	2.12
C	800	519.38	8129.12	1336.29	0.16	6.08	50.65	21.22	2.39

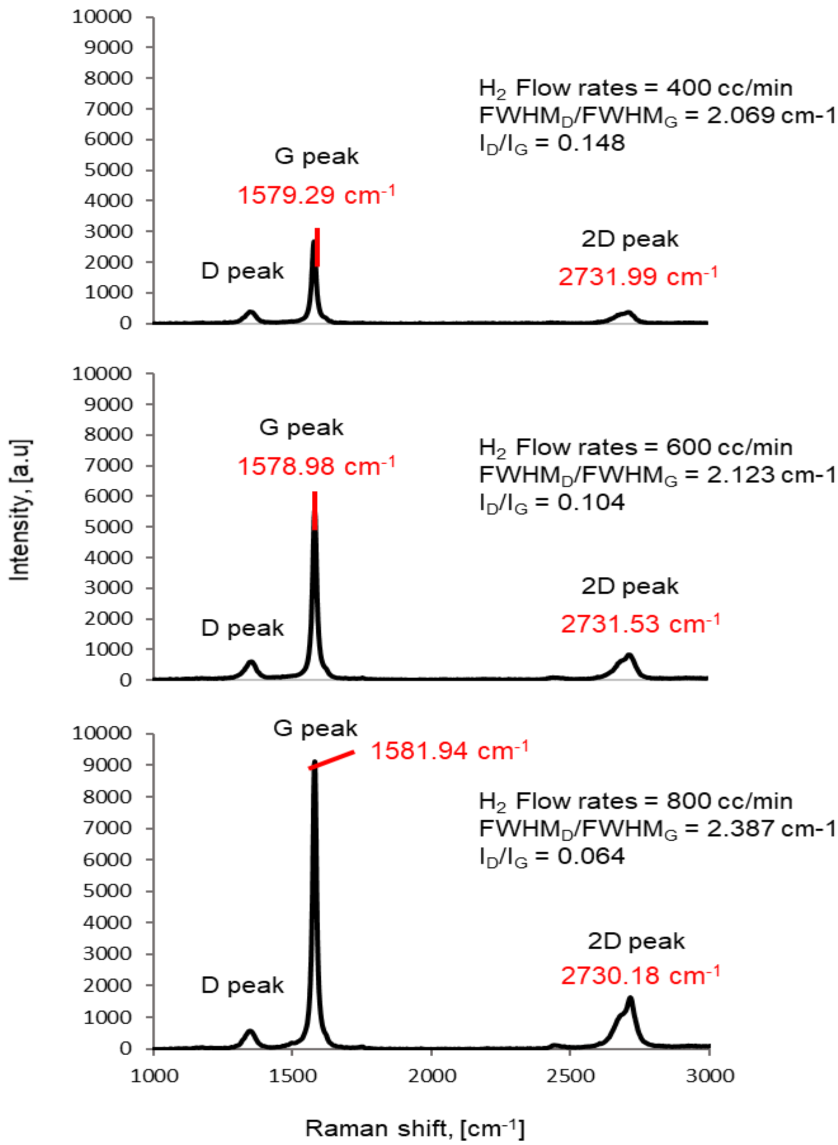

Figure 6: Raman spectra of synthesized graphene deposited on copper particles at various H_2 flow rates.

CONCLUSION

In summary, the uniform dispersion of surface 'wrinkles' observed using SEM and TEM indicates that graphene distribution on the surface of Cu particles is more homogeneous. The temperature and flow rate are critical in the CVD process for growing graphene. The quality of the graphene was improved with increasing hydrogen flow rates. When the hydrogen flow rate is raised, the higher carbon content on the Cu particle surfaces improves the quality of the graphene grown by having a low density of structural defects. Furthermore, the multilayer graphene was successfully synthesised on the Cu particles by CVD method and was successfully verified by using Gaussian analysis of Raman spectroscopy.

Owing to the excellent lubricating properties of graphene, further study into their synergistic impact as oil additive can be conducted in order to attain superlubricity properties.

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