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Electronic Textile from Lyocell and *Very Few Layer Graphene* : Studies and Review

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ABSTRACT

Electronic textiles (e-textiles) are generally made by coating fabrics with conductive particles to impart conductive and electromagnetic properties to textile fibers and filaments. E-textile can be made by several methods, such as "dip and dry", pad-dry, screen-printing, or injeksi printing. Lyocell is the latest generation of cellulose fibers that are used as textile raw materials. Lyocell has naturally hyperhydrophilic properties and greater moisture absorption. Then, graphene is a nanomaterial composed of carbon atoms with a hexagonal structure, it has a very high conductivity value, reaching 10^4 S/cm. Graphene can be produced in several forms, such as very few layer graphene (VFLG). This paper aims to improve understanding of the research and review of electronic textiles created by combining lyocell textile with very few layer graphene (VFLG). This composition can enable the formation of sustainable electronic textile composites.

1. Introduction

In Indonesia, Lyocell material is used for textile raw material needs for bed linen and clothing (fashion). Lyocell is the latest generation of cellulose fibers. The development of lyocell was driven by the desire to produce cellulose fibers that have a better cost and performance profile. Lyocell is a type of RCF (regenerated cellulosic fiber). RCF It is a type of man-made fiber made without changing the chemical structure of cellulose. Compared with other man-made fibers, such as synthetic fibers from synthetic polymers, all RCF raw materials are polymeric materials from nature (Woodings, 2001). Lyocell is made by dissolving non-derivative cellulose in organic and aprotic solvents. When compared to the manufacture of viscose fibers, lyocell has several advantages, namely the use of N-methyl morpholine oxide (NMMO)/H2O solvents that can directly dissolve cellulose, without the processes of mercerization, aging, xanthation and other treatments, so that the entire process is efficient and only lasts for 3–4 hours. In addition, the NMMO solvent is a non-toxic, environmentally harmless, and biodegradable chemical. Then, NMMO solvent after processing can be recovered at a rate of 98.5% to 99.0% and this makes lyocell a sustainable product.

Textile technology advancements are currently leading to the production of electronic textiles for a variety of applications such as smart clothes, IoT-connected sensors for healthcare, and others. The growth of electronic textiles (E-Textile) will expand in the next 10 years, with an estimated growth of up to 5 billion USD by 2032 (IDTechEx, 2022). The outcome of the E-Textile field will be an expansion in commercial products and the development of new techniques for incorporating electrical functions into textiles. Electronics textiles (e-textiles) are generally made by coating fabrics with metal particles such as copper, aluminum, or silver to impart conductive and electromagnetic properties to textile fibers and filaments. However, metallic materials have some disadvantages. For example, aluminum has a tendency to form an oxide surface that is resistant to electricity, which can cause overheating of the joints in smart textile filaments (Hughes-Riley et al., 2018). Another disadvantage of metal materials is that they can rust due to a chemical reaction between the metal and oxygen in the air.Metal materials can also cause stiffness that makes the textile uncomfortable to wear and ends up as e-waste.

The issues and concerns about metallic conductor materials, as well as the demand for clean energy use in new electronic devices, it can be one of the inputs used to develop other efficient conductor materials. One promising material that is currently being developed is graphene. Among various carbon materials, graphene is the most commonly used material as an electrode material because it has a very high conductivity value, reaching 10^4 S/cm (Zeng et al., 2014). In addition, graphene is a nanomaterial composed of carbon atoms with a hexagonal structure, so it has lighter, stronger, and more flexible properties. Graphene can be produced in several forms, such as very few layer graphene (VFLG), graphene oxide (GO), reduced graphene oxide (rGO), and graphene nanosheets (GNs). By combining Lyocell textile material with graphene, it can enable the formation of textile electronic composites. In addition to increasing the value of Lyocell, electronic textile products will be environmentally friendly, sustainable, renewable, softer, more elastic, washable, and will create a very practical and safe product for use in new technologies.

2. Materials and Methods of E-Textile

Fibers and filaments that can be used as e-textiles must have certain characteristics in order to achieve the desired specifications, namely sheet resistance value, conductivity value, tensile strength, and durability of graphene coating after filament washing. Shateri-Khalilabad and Yazdanshenas (2013) conducted research on coating cotton filament with graphene oxide (GO) through the conventional "dip and dry" method for electroconductive fabric manufacturing applications. The fabric that has been coated with GO is then immersed in a reducing agent solution to convert graphene oxide into rGO. The results showed that $Na_2S_2O_4$ is the best reducing agent, with a resistance value of 19.4 kΩ/cm and a reduction time of 30 minutes to form GO perfectly (Shateri-Khalilabad and Yazdanshenas, 2013).

Zhang et al. (2016) conducted research on the coating (layer by layer) of graphene oxide (GO) and reduced graphene oxide (rGO) on cellulose fiber using the multi-dipping-drying treatment method for the application of making flexible conductive devices. Graphene oxide with a concentration of 2 mg/ml was sonicated for 2 hours, then the filament was dipped in the solution for 30 minutes at room temperature, then dried at 50^{o}C for 12 hours. From the study, the results obtained a volume resistivity of 33.31 Ω cm in the flat filament state (weft direction) and 19.17 Ω cm when the filament is curved (warp direction) (Zhang et al., 2016).

Furthermore, Mengal et al. (2016) conducted research on the manufacture of electrodes using lyocells coated with graphene nanosheets (GNs) through the conventional "dip and dry" method. The resulting electronic textile has a very high surface conductivity with a very low surface resistance value of only 40 kΩ/sq, without using any binder or adhesive in the processing step. The lyocell used is a 100% lyocell plain woven fabric (Standard Tencel®) with a density of 140 g/m2 and a construction of 30 x 30 (Ne) or 93 x 75 inches, supplied by Lenzing AG, Austria.

Karim et al. (2017a) conducted research on the coating of graphene oxide (GO) and reduced graphene oxide (rGO) on cotton filament (cotton fabric) using the pad-dry technique method applied to the manufacture of bending sensors. The cotton filament was passed through a padding bath filled with rGO at a concentration of 3.2 mg/ml. The filament was then pressed with a roller to squeeze out the excess rGO. In the study, rGO formed a uniform layer around the fiber, with sheet resistance on the rGO-coated filament reaching 36.94 kΩ/sq after coating on the 5^{th} padding.

Then Qu et al. (2019) conducted research on graphene oxide (GO) coating in the form of patterns on viscose filament nonwovens using screen-printing and pad-dry-cure processes for application in conductive inks. The paste for printing ink was prepared by adding GO solids to water and stirring it for 24 hours. GO was controlled at a concentration of 18.5 mg/g. The results obtained for the sheet resistance of the filament were in the range of 1.2–6.8 kΩ/sq.

The use of GO has many disadvantages. GO is a derivative of graphene base material with properties of conductivity, strength, and flexibility that are lower than those of pure graphene base material. In addition, GO has many structural surface defects. Park et al. (2015) conducted research on the manufacture of strain sensors (sensors to measure strain) from yarns of rubber (RY), nylon-covered rubber (NCRY), and wool yarns (WY) coated with graphene nanoplatelets (GNP) using the layer-by-layer method. Before being coated with GNPs, the yarns were dipped in polyvinyl alcohol (PVA), which serves as a companion layer to form a stable and strong GNP layer. Then we continued with a polydimethylsiloxane (PDMS) coating, which serves to avoid the possibility of delamination of the GNP layer from the surface due to high strain.

Research by Liu et al. (2017) made a composite sensor from cotton textile coated with graphene oxide (GO) for heart rate sensor applications. The surface of this graphene cotton composite is coated with polydimethylsiloxane (PDMS), which serves as the abrasion resistance of the film, increases the ability to adapt to environmental changes, and extends the life of the fabric while maintaining the flexibility and comfort of the composite when used. Meanwhile, in the research of Kumar et al. (2020), the cotton fabric used for the Covid 19 virus mask material is coated with graphene oxide (GO) through the vacuum filtration deposition method. Furthermore, the GO fabric is coated again with polyaniline (PANI) through a chemical polymerization process. The results showed that the fabric can protect against UV radiation and that its electrical conductivity is higher when compared to cotton fabric without a graphene coating. They also reported that the fabric coated with GO and PANI still worked efficiently after 10 water washes.

There are many type of graphene materials that can use to make e-textile prototype such as graphene oxide (GO), reduced graphene oxide (rGO), and graphene nanosheets (GNs). The other types of graphene that could possibly be used to manufacture electronic textile is Very Few Layer Graphene (VFLG). According to Bazylewski & Fanchini (2019), very few layer graphene (VFLG) has similar characteristics to pure single-layer graphene. Research by Amri et al. (2021) mention that a simple and ecologically friendly two-step shear exfoliation (TASE) approach in rotating-blade mixers (kitchen blenders) and in high shear exfoliation (SE) in high shear rotor–stator mixer has successfully created a defect-free graphene with 1-3 layers called very-few-layer graphene, or VFLG. High-quality VFLG made this method a promising method for mass manufacture of high-quality graphene, and it was simple, affordable, and ecologically sustainable.

3. Component of E-Textile

Electronic textiles can be made by applying carbon-based inks that have conductive properties to the surface of textiles. These carbon-based inks or conductive inks consist of several constituent components, namely solvents, binders, surfactants, and carbonaceous materials as fillers such as graphene, carbon nanotube (CNT), carbon black (CO), dan activated carbon (AC). These components are needed to form the rheology of the ink, suspend, and stabilize the carbon particles in the liquid solvent to improve the ability of the ink sticking process on textiles (Khair et al., 2019).

3.1 Conductive Ink

Conductive ink is a thick thermoplastic paste that can conduct electricity by adding conductive materials during the manufacturing process. The function of conductive ink is to create conductive paths to be used as interconnect paths. Conductive ink is a multi-component system that contains conductive materials in liquid form. Basically, the ink is defined by a mixture of water, pigments or colorants, humectants, and other additives such as rheology and surface tension modifiers, binders, and defoamers. These additives make it possible to optimize the performance of conductive inks. In a more specific sense, conductive inks are suspensions of conductive nanomaterials in either water or solvent media with the addition of surfactants or polymers that act as stabilizers. This solvent must evaporate quickly after the ink has settled on the substrate surface but not dry quickly on the press when not in use. To obtain high electrical conductivity, ink manufacturers usually use conductive nanomaterials. The size of these nanomaterials must be smaller than the size of the pores or nozzle of the printing device to avoid clogging.

The ideal conductive ink should be cheap, easy to prepare, and have good printability, low viscosity, good stability, good adhesion to the substrate, and high electrical conductivity after printing and post-printing processing.When viewed based on the printing process of conductive inks, there are several factors that must be considered when formulating conductive inks. These factors are the selection of printing methods and the selection of conductive materials. Apart from these factors, Yang & Wang (2016) reported that conductive material-based inks should be stable against aggregation and settling to provide the best performance when produced. Conductive material-based inks tend to agglomerate, and this agglomeration can cause an increase in ink viscosity, leading to changes in ink properties and the clogging of pores and nozzles on the device during printing. Agglomeration of these conductive materials can be prevented by incorporating additives into the ink, such as dispersants, surfactants, adhesion promoters, stabilizers, and others.

As is known, conductive material-based inks are suspensions of conductive materials in additive-based media. The volume of conductive material is generally maintained above the surface of the substrate, and thus there is a minimum volume required to achieve connectivity between particles. According to Kamyshny & Magdassi (2014), the characteristics of good conductive inks are good dispertion to solvent, printability, good adhesion with the substrate, high resolution, minimal printer maintenance, and a long shelf life. However, physical properties such as viscosity and surface tension are dominate the conductive ink properties during the printing process and the quality of the printed conductive patterns.Viscosity is a fundamental characteristic of all liquids and a parameter used to measure the resistance of liquids when flowing or deforming when shifting. This resistance is caused by cohesive intermolecular forces, which give rise to friction between adjacent layers from the inrelative motion of the fluid.

Many parameters can affect viscosity, including temperature and pressure. These two parameters are considered to be one of the critical properties of printing ink. The viscosity value will decrease when the temperature increases, and vice versa.According to Ihalainen et al. (2015), the ideal conductive ink viscosity value is in the range of 1–20 mPa; this range serves to avoid problems during the printing process, including clogging of printing nozzles, clogging of the pores of the printing device, and satellite or double droplets. If the conductive ink is too thick, it will cause the flow frequency to decrease due to the decreased absorption rate. Meanwhile, conductive inks that are not viscous enough can cause the flow frequency to be too high, resulting in the release of unstable ink droplets. This can also be observed in Figure 2.5, where the viscosity value decreases with increasing temperature. In the research of Dybowska-Sarapuk et al. (2018), the viscosity value of graphene ink at room temperature is between 7.5 mPa and 10 mPa, this value is considered acceptable for use in the printing process on fabric.

3.2 Solvent

An important component of conductive inks is the solvent. The function of the solvent is to facilitate ink flow and act as a transfer medium for the conductive material from the ink to the substrate. Solvent selection depends on the binder, conductive material, and fabrication method used. The ideal solvent for a particular binder and active material is selected based on its solubility parameters and vapor pressure. Generally, solvents with low vapor pressure are suitable for screen printing ink application technology due to their high degree of stability.Screen printing inks take relatively longer to process on the fabric surface than other processes. On the other hand, solvents with high vapor pressure are not suitable due to their tendency to volatilize at normal temperatures.

On the other hand, volatile solvents or solutions with high vapor pressure are more effectively used for inkjet printing technology. Therefore, water, ethylene glycol, methanol, and isopropanol are widely used solvents for the formulation of printing inks for carbon-based materials. These water-solvent conductive inks are preferred because the conductive material will require more time to penetrate into the substrate (textile fiber). According to Kim et al. (2017), the selection and number of components in the ink will depend on the type of fabrication technology and on what type of electronic media will be printed. An important component of conductive inks is the solvent, which is useful for facilitating ink flow and transferring conductive materials from the ink to the substrate. The solvent selection depends on the binder, activated carbon material, and fabrication method. The ideal solvent for a particular binder and active material is selected based on their solubility parameters and vapor pressure (Kim et al., 2017).

3.3 Binder

The value of electrical conductivity in textile electronics will be obtained from Very Few Layer Graphene (VFLG) nanoparticles; this value is formed due to the presence of pathways for electrical transport consisting of charge transfer over short distances (Shin et al., 2021). However, this can lead to aggregation and create holes in the fabric's polymer matrix network. The choice of biders for the preparation of conductive inks is also very important, and it depends on the solvent and its interaction with the substrate. Coatings serve to improve rheological flow properties and provide viscosity that can facilitate the ink absorption process. Conductive polymers can be used as binders to improve the electrical properties of the textile electronic pattern to be printed. Frequently used binders are bovine serum albumine (BSA), polydimethylsiloxane (PDMS), polyvinyl alcohol (PVA), calcium silicate (Ca_2SiO_4), and polyaniline (PANi). From those binders on this section we choose polyvinyl alcohol (PVA) and calcium silicate (Ca_2SiO_4) for further discussion.

According to Kohlpaintner et al. (2000), polyvinyl alcohol is a highly hydrophilic synthetic resin made by polymerization of vinyl acetate followed by partial hydrolysis of the ester with a base catalyst. Polyvinyl alcohol, which is widely found in the form of white to cream-colored granules or powder, has a pH of 5-8 (when dissolved at a concentration of 4%). Polyvinyl alcohol (PVA) is a water-soluble polymer; its characteristics can form a thick and smooth film after its water content evaporates at high temperatures. Bingham et al.

(2002) mentioned that PVA is widely used in the coatings sector (for example, as a thickening agent for aqueous systems such as inks), in adhesives, for finishing on paper and cardboard, for paper coatings, as a binder for peelable coatings, and as a protective colloid for dispersions.When manufacturing textile electronics, PVA will be attached to the textile surface through non-covalent bonding interactions that include hydrogen bonding, van der Waals forces, and hydrophobic attraction forces (Park et al., 2015). The use of PVA for adhesives in textile electronics can be compatible with most textiles and is environmentally friendly due to its low volatile organic compound (VOC) release properties (Yang et al., 2014).

While calcium silicate ($Ca2SiO_4$) is one type of resin that can dissolve in water, Chandra et al. (2016) mentioned that calcium silicate ($Ca2SiO_4$) can be used as a silicon doping agent when making ink. Silicone-coated inks help to reduce and prevent scratching of printer print head components.In addition, in the research of Peng et al. (2019) mentioned in the ink manufacturing process, calcium silicate material functions as a filler or filler material that can act as a substitute for pigment parts, reduce pigment costs, and can also function to adjust ink properties, such as liquid thickness, and can increase ink flexibility. When manufacturing electronic textiles prototype, calcium silicate (Ca_2SiO_4) will be added to the conductive ink as a filler or filler of textile pores and act as a customizer of ink properties, such as thickness, fluidity, and flexibility. Peng et al. (2019) mentioned the combination of ink with calcium silicate and volatile organic compounds, showing a reduced amount of evaporation of substances into the atmosphere. This shows control over the reduction of ink VOCs and makes the ink with calcium silicate (Ca_2SiO_4) an environmentally friendly coating.

3.3 Surfactan

In addition, another key component of conductive inks is surfactants, which are used to disperse the active ingredients evenly and prevent agglomeration of carbon particles by repulsion due to electrostatic forces. Surfactants play an important role in imparting wetting characteristics to the ink on the substrate surface. In addition, surfactants have an effect on functional ink device technology, where inks can be applied to less absorbent substrates such as coated paper and non-absorbent substrates such as textile fibers or films. In the journal Htwe & Mariatti (2022), it is mentioned that when making graphene conductive ink, it will be very difficult to disperse into solvents. This is due to the hydrophobic character of graphene carbon. Surfactants are thus used as dispersing agents to improve graphene dispersion in conductive ink via van der Waals forces, hydrogen bonding, electrostatic activity, and - interactions.

The presence of electrons on graphene in the ink solution can make the surface of the graphene layer negatively charged. Therefore, surfactants also serve to enter between the graphene layers, thereby increasing the possibility of agglomeration. Various surfactants, such as ionic, non-ionic, polymeric stabilizers, and bio-surfactants, can be widely preferred choices for improving ink stability. According to Kamyshny (2011), the viscosity value of conductive ink should be in the range of 8–15 cP. This range of values is intended to allow the conductive ink to pass smoothly through the nozzle without experiencing problems related to leakage, drying, or coagulation.

4. Conclusion

Lyocell is the latest generation of cellulose fibers. Because it is made by dissolving non-derivative cellulose in organic and aprotic solvents such as N-methyl morpholine oxide (NMMO), which can be recovered at a rate of 98.5% to 99.0% after processing, it is classified as a sustainable product. In addition, graphene is a nanomaterial composed of carbon atoms with a hexagonal structure, so it has lighter, stronger, and more flexible properties. Graphene is the most commonly used material as an electrode material because it has a very high conductivity value. There are many type of graphene materials that can use to make e-textile prototype such as graphene oxide (GO), reduced graphene oxide (rGO), and graphene nanosheets (GNs). The very few layer graphene (VFLG) is another types of graphene that could possibly be used too. High-quality and ecologically sustainable VFLG made by the two-step shear exfoliation (TASE) and shear exfoliation (SE) methods. This paper is aimed at better understanding the method of electronic textile made from lyocell and very few layer graphene (VFLG). By combining lyocell textile with very few layer graphene (VFLG), it can enable the formation of sustainable electronic textile composites. To create electronic textiles from lyocell and very few layer graphene, one of several methods can be used: "dip and dry," pad-dry, screen-printing, or injeksi printing.

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