Chemical Surface Modification of Oil Palm Fibre: a Case study of developed Oil Palm Fibre-Plastic Composites

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ABSTRACT - Application of chemicals to treat the surface of oil palm fibre could have effects on the characteristics of plastics when oil palm fibre and plastic composites are developed. Identification, sampling, and review of literature relating to oil palm fibre (OPF) chemical surface treatment, vis-a-vis the formation of OPF-plastic composites were done in this study. Chemicals relating to oil palm fibre surface treatment and their effects on OPF-plastic composite characteristics were analyzed. Some surface chemical modifiers influence specific characteristics of composites positively and better than others, while the same chemical may have influence negatively on other traits. Thus, the degree of effect of chemical treatment of OPF-plastic composite could depend on the interesting characteristics, type of chemical, the quantity of chemical and type of plastic.

KEYWORDS: Chemical surface treatment; Composite materials; Oil palm fibre; Plastics; Silane treatment

1. Introduction
The composite material formation is made feasible and stabilized when there is better and strong interfacial adhesion between the component materials [1]. In the case of vegetal fibre and polymer composite formation, with fibre acting as the filler material and polymer as a matrix, the presence of silica bodies on fibres, the presence of hydroxyl molecules in cellulose fibre, and the presence of oil residue on fibres negate the recognition of this objective [2]. Nevertheless, techniques to improve fibre-polymer adhesion and stability, including physical, biological, and chemical methods are available [3, 4]. However, the chemical treatment of fibre is the most efficient and effective of late [5]. Oil palm fibre (oil palm mesocarp fibre (OPMF) or oil palm empty fruit bunch (OPEFB) fibre) as a filler material has become popular as a result of availability and feasibility, particularly in regions where climatic conditions for the plant are favourable [6]. These regions include Asia, West, and Central Africa, and South America. The purpose of the chemical treatment of vegetal fibres to expose the responsive and reactive functional groups found with fibre surface to introduce hydrophobic properties of cellulosic fibre; and subsequent compatibility within the composite microstructure. Manifold types of chemicals are available in this regard. Nevertheless, the following are reliable: alkalization; silanization; acetylation; benzoylation; permanganate treatment; peroxide treatment; isocyanate treatment; and maleated coupling [1-7].

The presence of alkali diminishes cellulose, hemicellulose, lignin pectin, and wax contents of vegetative fibre. Alkalis can depose part of cellulose and hemicellulose, in particular, to
disable, to a large extent, the hydrophilic property of the fibre. Silane treatment also acts as a coupling agent, through a bridging effect of the silands, between the matrix functional group and the hydroxyl class ensuring a strong continuous linkage within the hydroxyl and matrix functional groups. The purpose of the acetyl group is to render the fibre-matrix composite more stable regarding hydrophobicity. Though acetylation is reliable, it is better efficient when it is combined with alkalization or anhydride treatment, i.e., after acetylation has been introduced. Peroxide treatment is similar to benzylation and acetylation. They require pre-treatment such as superheating, enzymatic treatment, alkalization, or a similar treatment before introduction, to be more productive. In achieving the desired objective free radicals are introduced to disorganize the hydroxyl group in fibre to achieve stronger interaction between matrix and fibre interface. Permanganate solution also disorganizes the hydroxyl group by acting as a graft copolymerization of cellulosic fibre and therefore enhances the interlocking chemical characteristic of composite microstructure.

Isocyanate treatment improves the interaction between matrix and fibre by influencing both cellulose and lignin through reactions between the -OH presence in cellulose and lignin on one hand and urethane group on the other. Thus, the introduction of anhydride groups also forms an interlocking linkage between hydroxyl groups of fibre and the anhydride group of the coupling agent. A protective or surrounding blanket may also be formed surrounding the fibre, and further add to the strength of fibre and matrix interfacial bond. Aside from permanganate, graft polymerization agents include vinyl (CH2=CH), acrylic acid (CH2=CHCOOH), and acrylonitrile (CH2=CH-C≡N) agents. In the process, free radicals such as carboxyl (COOH) and nitrile (C≡N) are released to interact with the hydroxyl group in cellulose to ensure stronger intermolecular bonds. Peracetic acid ensures delignification to a high degree, and consequential fibre stiffness, rendering composite more brittle. The purpose of this review paper is to examine empirical studies relating to chemical modification of oil palm fibre (OPF)-plastic composite formation. The objective, however, is to identify the type of OPF-plastic blends, surface-modifying agents applied in research, and their influence on specified mechanical, physical, thermogravimetric, and water absorption characteristics on OPF-plastic composites.

2. Literature Review 2.1 Oil palm fibre-polypropylene blend

Chemical treatment of oil palm fibre could have effects on oil palm fibre-polypropylene (OPEFB fibre-PP) composites. For example, in one study, the addition of Maleic anhydride-polypropylene (MAPP) affects both impact strength and thermal degradation Nasution et al. [1]. Trimethylolpropane tricrylate (TMPTA) also influenced the mechanical properties of PP-cellulose composite significantly. Polypropylene is a thermoplastic material. While TMPTA significantly enhanced the toughness of OPF-PP and PP-cellulose composites, PP-cellulose properties were better improved since tensile strength increased to 43MPa, impact strength to 43J/m and flexural modulus to 3.3GPa when 2% TMPTA was incorporated. It was also reported that at 15% chemical loading of Maleic anhydride (MAH), 40% OPF loading and 60 mesh size fibres; OPF-PP composite flexural strength, flexural modulus, flexural toughness and impact strength of 40MPa, 4GPa, 7kPa, and 80J/m were respectively exhibited. Without chemical treatment, there was weak interphase bonding. This weak bonding caused a partial separation between filler and matrix, yielding micro-spaces, and consequential stress propagation during impact and thus improving brittleness [8]. Impact resistance was therefore weak, unlike when MAPP was incorporated into the composite [1]. Due to the reaction between the MAH group and the hydroxyl group of cellulose and hemicellulose in OPF [9]. The ester groups so ensued exposed the polyolefin chain of MAPP on the fibre surface, causing PP in MAPP to diffuse into the polypropylene matrix. The
consequential interaction produced linkages between filler and matrix interface [10]. In other words, enhanced mechanical properties of composites, whereas a result of covalent bonding between MAH and PP. Compatibility between non-polar PP and polar group (OH) of OPF was invoked as a result of MAH treatment [11]. However, the lower molecular weight of MAPP as compared to that of the matrix (PP) could lead to a plasticizing effect and reduced impact strength when MAPP was applied in relative excess [9].

In another study, when residual oil was removed from the OPF surface, both tensile strength and toughness of OPF-glass fibre-PP hybrid composite could be improved significantly [12]. Tensile strength and toughness increased from 7MPa and 50kPa to 13MPa and 130kPa, respectively, when fibre surface oil was removed. Despite this, there was no significant change in tensile modulus (700MPa) and elongation at break (2.3%) when resin was removed. Upon removal of residual oil, however, flexural strength, flexural modulus, and flexural toughness increased significantly from 13MPa, 1.9GPa, and 5.5kPa to 27MPa, 2.3 GPa, and 26kPa, respectively. Flexural and tensile characteristics of OPF-glass fiber-PP hybrid composite were further increased when coupling agents were applied to OPF. Comparatively, maleic anhydride-modified PP (Epolene, E-43) treated composites exhibited better properties than 3-(trimethoxysilyl) polypylmethacrylate (TPM), or polymethylene (polyphenyl) isocyanate (PMPPIC) treated ones [13]. Treating OPF with Epolene E-43 could reduce the hydrophilic capacity of OPF-PP composites in terms of E-43 presence and quantum for two reasons: one; the PP chain presence in MAPP, and two; the blockage of the bond between the hydroxyl group and water due to the presence of MAH residue of MAPP to react with OPF hydroxyl groups [11]. Thus OH was hindered and obstructed by MAH to be released to bond with water molecules, consequently rendering the composite hydrophobic. The PP in MAPP directly bonds with the PP matrix.

The addition of MAPP to OPF also improved the thermal stability of OPF-polypropylene composite. Thermogram analysis showed that weight loss was 100% when MAPP was absent. However, the presence of 6 and 8%wt MAPP reduced weight loss to 90% thus slowing down thermal degradation rate due to interfacial linkages or bonds between filler and matrix; caused by the diffusion of PP in the MAPP into the matrix (waste PP) to cover the surface of OPF fibres. In other words, the effect of heating is resisted due to bridge formation between fibre and polypropylene matrix. It was, however, observed that there was a small increase in thermal degradation temperature getting to the end of the process through MAPP was present. This may be due to the presence of charred layers, which acted as insulation to further degradation [1].

### 2.2 Oil palm fibre-polyurethane blend

At 40% OPEFB fibre loading and NCO/OH (cyanate ion/hydroxyl ion) ratio of 1:1, the tensile strength of oil palm fibre-polyurethane (OPF-PU) OF 26MPa was exhibited. When OPF was further treated with toluene diisocyanate (TDI), tensile strength increased to 30MPa. Nevertheless, the tensile strength of composite reduced to 28MPa when hexamethylene diisocyanate (HMDI) was employed to treat OPF. Thus the treatment of OPF with TDI and HMDI helped increase the tensile strength of OPF-PU composite through enhanced interfacial filler and matrix linkages [14].

### 2.3 Oil palm fibre-polyvinyl chloride blend

Benzoylation treatment of OPEFB fibre had a positive influence on the mechanical properties of oil palm fibre-polyvinyl chloride (OPF-PVC) composites due to improved adhesion between fibre and PVC (a thermoplastic material) at the molecular level [15]. Impact strength, tensile strength, and stiffness were impacted in this regard. In another study, at 10% fibre
loading, methyl acrylate (MA), which was grafted on OPF, increased the ultimate tensile strength (UTS) of polyvinyl chloride-epoxidized natural rubber (PVC-ENR) from 8MPa to 10MPa [16]. The influence of grafting was also positive for elongation at break. However, grafting with MA harmed flexural modulus, impact strength, and hardness. The addition of an acrylic impact modifier also reduced both strength and modulus of OPF-PVC composites [17].

2.4 Oil palm fibre- polyester blend

The interfacial shear strength in OPEFB fibre-polyester can be significantly improved by treating OPF with chemical modifiers such as sodium hydroxide (NaOH) [18]. Alkalis tend to expose the fibre surface to the polyester matrix better. It is also able to create pits on the re-fibre surface. Polyester, a thermoplastic, is, therefore, able to penetrate the filler bundles to enhance fibre-matrix mechanical bonding and improved interactions between the two. Acetylene also can reduce the number of voids in composites, thereby improving the wettability of fibre. This has a positive effect on the impact strength of the fibre [19]. Titanate modification of fibre surface could decrease the tensile stress of composite, while both silane and acetylation modification could lead to a slight increase [20]. Acetylation also increased flexural modulus significantly while alkali treatment enhanced abrasion resistance. Thus generally, untreated fibres improved adhesion resistance of pure polyester resin by 50 and 60%. However, treating fibres with alkali, silane, or acetylene could improve adhesion resistance by 75-85%.

Water-absorption characteristics were improved upon acetylene treatment of fibres [19]. At 100°C, water-absorption property of OPF-polyester reduced from 15.8% (untreated) to 5.7% (acetylene-treated). This was attributed to better interfacial bond between filler and matrix, which positively influenced hydrophobic characteristics of the fibre and the resulting composite. Chemical treatments of OPF could preserve the mechanical characteristics of OPF-polyester composites with longevity [19]. Aging in de-ionized water was lower in terms of tensile stress, tensile modulus, and elongation at the breath when fibre was chemically treated. However, aging in terms of tensile stress and modulus increased in the first 3 months when acetylation was utilized, though the rate generally reduced with time. Exposure of composite to the soil after many months did not affect the aging of chemically treated composites as compared with untreated ones, with the decrease in loss of strength of titanate greater than silane and acetylene in that order [20]. Chemical treatment of fibre significantly reduced the mass degradation of OPF-polyester composites when subjected to weathering [21]. Mass degradation was highest for acetylene, followed by silane and titanate treatment in that order.

2.5 Oil palm fibre-polyethylene blend

Ewulonu and Igwe [9] have observed that the properties of oil palm empty fruit bunch (OPEFB) fibre-filled high-density polyethylene HDPE), a thermoplastic, could be influenced by the application of maleic anhydride (MA). Acting as a compatibilizer, the inclusion of MA between 1 and 5% by weight caused a general increase of hardness of the high-density polyethylene composite. The study concluded that the addition of MA increased the interfacial bonding between OPEFB filler and HDPE matrix through microstructure modification of the surrounding matrix. Similarly, the presence of MA influenced the specific gravity of composite. The specific gravity of composite increased due to the addition of MAPE. This was because MA enhanced the adhesive bond and interaction between OPEFB filler particles and polymer matrix, in agreement with the work of Yu et al. [22]. The presence of MAPE reduced the hydrophilic and flame propagation characteristics of OPEFB-HDPE composites. Reduction in water sorption characteristics was attributed to the reaction between MAPE and –OH group to form hydrophobic ester linkages. This result was found to
agree with the findings of Chollakup et al. [23]. With reference to flame propagation, the study observed that the application of MA content appeared to decrease the combustion initiation energy of product composite, thus retarding the flame propagation process.

The addition of MA up to 0.125wt saw a perpetual increase in tensile strength, after which weight-related increase in tensile strength was virtually non-existent. It was believed to be as a result of the reaction between MAPE molecules and hydroxyl groups of cellulose and hemicellulose present in OPEBF, forming hydrophobic esters (reaction between the matrix phase and the reinforcement phase). Compatibility between long continuous chains in MAPE molecules and polymer matrix chains was also able to cause physical entanglements between reactants. Thus when MAPE is present, there is both physical and chemical bonding within the composite leading to higher tensile strength [9]. This means that beyond 0.125wt% of MAPE, stress transfer from matrix to fibre was much lower.

Similarly, the addition of MAPE significantly improved elongation at break (EB) of OPEFB-HDPE composites when MAPE content was between 0.625 and 0.75wt%, beyond which there was a decrease in EB. The initial increase was explained to be a result of increased adhesive forces between the matrix (polyethylene) and filler (OPEFB). The reduction observed when MAPE was beyond 0.75wt was explained to be due to the migration of profound MAPE around OPEFB, which resulted in self entanglement within MAPE itself instead of polymer matrix causing slippage [9]. A study by Chollakup et al. [23] on the title “Polyethylene green composites reinforced with cellulose fibres (coir and palm fibres): effect of fibre surface treatment and fiber content,” found that oil palm fibres have fewer impurities than coir fibres. Alkali and acid (50% hydrogen peroxide) treatments were separately applied. The study observed weaker interaction between the LDPE matrix and fibre, when alkali was administered, resulting in fibre pullout from matrix and traces of holes. However, bleached fibre using hydrogen peroxide as a bleaching agent yielded composites with stronger interaction between fibre and matrix. Thus MAPE, alkaline, or hydrogen peroxide treatment of OPF could influence the characteristics of OPF-LDPE composites.

2.6 Oil palm fibre polyester/polyurethane blend

The effect of silane-treated oil palm mesocarp fibre (OPMF) on hybrid polylactic acid/polycaprolactone/nano clay (PLA/PCL/NNC) composite has been documented by Eng et al. [22]. Polylactic acid and polycaprolactone are thermoplastics. The study observed that though 85% PLA and 15% PCL composite has the best tensile properties (1%wt clay) in terms of tensile strength, elongation at break and tensile modulus (40.45, 0.95 and 865.5 respectively); addition of 10% methacrylate silane-treated mesocarp filler (fibre) increased tensile strength by 11.37% from 36.32MPa to 40.45MPa. The treated fibre was found to be more hydrophobic than unmodified OPMF below 10% silane content. Compatibility between fibre and matrix was also found to be enhanced, resulting in increased strength, stiffness, and interfacial adhesion. Beyond 10% fibre loading, tensile strength decreased, though silane was present because of more fibre weakens the intermolecular attraction. In addition to this, there was weak bonding between hydrophilic filler and hydrophobic polymer matrix, thus obstructing stress propagation between hydrophilic filler and hydrophobic matrix. At 10% silane treatment, tensile modulus increased from 685.8 MPa to 865.5 MPa. This was explained by the fact that there was an improvement in fibre/matrix attraction resulting in improved stress transfer within the composite. Improved fibre/matrix adhesion similarly improved elongation at break to a value of 0.95mm.
In addition to these findings, flexural properties of silane (10%) treated OPMF also increased as compared to untreated composites, from 36.53MPa to 42.96MPa (17.50% increase). The study suggested better adhesion between matrix and fibre since silane acts as a bridge between the two when they are in contact.

The study further observed that the flexural strength of silane treated OPMF composites was higher than the tensile strength of composites as a result of fibre orientation on the surface of the composites. However, when fibre loading was increased, flexural strength reduced even when fibre was treated with silane. This was explained due to self-entanglement as a result of the overpopulation of fibre and the consequential struggle for space. The presence of 10% silane treated OPMF similarly increased flexural modulus from 2437MPa to 2758MPa, an increase of 13.1%. It was explained by the fact that there was more compatibility between less hydrophilic fibre and matrix, resulting in efficient and better stress transfer in the continuous interface.

The Izod impact strength of composites was additionally enhanced due to the presence of 10% silane treated OPMF. The highest impact strength improved from 95.44J/m (unmodified) to 141.66J/m (modified); about 48.5% increase. The study indicated the reason as the hindrance of deformation and ductile mobility of polymer molecules by fibre, reducing the composite's ability to absorb energy during crack propagation. It further indicated that when OPMF is treated with silane, its presence enhances fibre wettability resulting in the creation of less crack initiation and fewer voids within the composite with fewer flaws and consequently enhanced impact strength. Thermogravimetric analysis (TGA), however, indicated no significant effect on composites when OPMF was treated with silane. The onset temperature was set at 217.67°C. The study attributed this result to a low amount of fibre addition (30%) to composites. The study concluded that silane treatment is not a factor to consider when it comes to the thermogravimetric properties of the composites.

Comparatively, silane treated OPMF hybrid composites showed lower water absorption than untreated composites. At the initial studies, all samples exhibited sudden increases in water absorption, remaining constant and 5.29% for silane treated OPMF hybrid composites and unmodified composites, respectively, obeying the Fickian diffusion behaviour. Fick's law states that the mass of water absorbed by a body increases linearly with the square root of time and then gradually slows until the equilibrium plateau is reached. The study explained the behaviour of silane treated OPMF hybrid composites, and modified fibre is less hydrophilic. The number of hydrophilic hydroxyl groups reduces by reacting with silane, which results in water exclusion from the substrate. Scanning electron microscopy (SEM) showed excellent adhesion of silane treated OPMF hybrid composites within the interphase showing the bedding of fibre in the matrix. The surface cavity was found to be absent, indicating the absence of fibre pull out during tensile experimentation, thus indicating excellent fibre/matrix adhesion [24].

2.7 Oil palm fibre (OPEFB) and epoxy blend
A comparative study regarding surface treatment of oil palm fibre (OPEFB) and epoxy (EP) (thermoset, requires curing) composites confirms that fibre surface modification can be effected using a silane coupling agent, acetic acid, or alkaline solution [26]. The tensile strength of untreated fibre composite (11.48MPa) increased to 12.22MPa, 13.88MPa, and 15.83MPa through alkaline, acetylation, and silane treatment, respectively. Similar increases also occurred to the tensile modulus. While the tensile modulus of untreated fibre composite was 1342MPa, it increased to 1708MPa, 1726MPa, and 2582 MPa through acetylation,
alkaline, and silane treatments respectively. However, elongation at break reduced from 4.09% for untreated fibre composite, to 3.9%, 2.53%, and 2.24% through acetylene, alkaline, and silane treatments, respectively. Generally, chemical treatments tend to increase composite stiffness as compared to untreated fibre composites.

Scanning electron microscope (SEM) image of alkaline treated composites, when tensile fractured, indicated thin sheared fibres. Fibre diameter was reduced while the surface area of contact between fibre and matrix was increased, thus enhancing bonding behaviour. The application of silane showed broken fibre ends instead of pulling out characteristics. Fibre end was characterized by cracks, showing prodigious breakage instead of pull out. This was an indication of enhanced interfacial adhesion.

Additionally, silane treatment ensured surface roughness improvement, which yielded higher mechanical interlocking between filler and matrix. The coupling characteristics of silane were thus exhibited in this regard. Similarly, the SEM image showed significant adhesion between fibre and matrix. Surface morphology was exhibited of the fibre-matrix composite [25, 26, 27].

Alkaline treatment improves the mechanical properties of consequent composite, such as tensile strength. In contrast, silane treatment had a significant influence on the interlaminate shear strength of composites, thus enhancing the tensile and flexural properties of fibre epoxy composites [28]. Silane is also most effective in enhancing hydrophobic characteristics of epoxy composites in humid environments. On the other hand, acetylation improves adhesion between fibre and matrix and further enhances the fracture properties of fibre-matrix composites [24].

### 2.8 Oil palm fibre surface treatment and their effects on OPF-plastic

Chemicals relating to oil palm fibre surface treatment and their effects on OPF-plastic composite characteristics were analysed and the following were observed: (1) Maleic anhydridemodified PP (Epolene, E-43) treated composites modified properties are better than 3(trimethoxysilyl) polypylmethacrylate (TPM) or polymethylene (polyphenyl isocyanate) (PMPPIC) for oil palm fibre-Polypropylene (OPF-PP) composites (2) Benzoylation treatment of OPF had a positive influence on impact strength, tensile strength, and stiffness of oil palm fibre-polyvinyl-chloride (OPF-PVC) composites; (3) Alkali, Titanate and Acetylene could influence water resistance, impact strength, tensile strength and decomposition of oil palm fibre-polyester (OPF-PS) composites (4) MAPE, alkaline or hydrogen peroxide treatment modified the characteristics of OPF-LDPE composites; (5) Addition of methacrylate silane increased strength, stiffness, impact strength, flexural properties, water resistance and general interfacial adhesion of Oil palm fibre-polyester/polyurethane (PLA/ PCL/NNC) composites; (6) For Oil Palm Fibre-Epoxy (OPF-EP) Composite, the alkaline treatment improved mechanical properties of composites such as tensile strength; silane treatment enhanced tensile, flexural and hydrophobic characteristics; while acetylation improved fracture properties of fibre-matrix composites

### 3. Conclusion
The abundance of OPF as waste material and other polymer waste, and the attempt to deal with environmental pollution plus poor sanitary conditions, has called for the application of OPF and plastic waste in the development of composite materials. The major challenge of these composite development has been fibre-polymer incompatibility and disaggregation arising from the –OH functional group in fibre.
Nevertheless, applying chemicals to fibre body surface has been found to reduce these challenges. Specific polymers that have been studied in this regard include Polyethylene, Polypropylene, Polyvinylchloride, and polyurethane. Others include Epoxy, Phenol formaldehyde, and Polyester. Contextually, particular chemicals that have proven their worth include maleic-anhydride polypropylene (grafting polymerization); toluene diisocyanate, hexamethylene diisocyanate; benzyl chloride; sodium hydroxide acetylene potassium permanganate (graft polymerization); propionylation; methacrylate silane (silanization); acrylonitrile (acrylation); hydrogen peroxide, dicumly peroxide and peracetic acid (acidification as bleaching agent). Nevertheless, NaOH (alkali), methacrylate silane (silane), and peracetic acid (acidification) appear to prove more useful modifying fibre surface. Alkalization and silanization better improve tensile, flexural, and hydrophobic characteristics, while acidification enhances brittle properties. Graft polymerization could cause an increase in hardness, specific gravity, and water resistance abilities. Enhancement of characteristics by a chemical may cause adverse effects on other properties. Applying more than one chemical also appears to influence specific characteristics more than others. A combination of two or more filler materials may optimize the enhanced characteristics of the developed hybrid composite material.

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