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Grafting of acrylonitrile onto allylated Plantain pulp by potassium-permanganate/toluene redox pair

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Abstract: The graft copolymerization of acrylonitrile onto allylated plantain pulp fiber by permanganate/toluene redox system has been investigated. Plantain fiber (Musa spp) was converted to the holocelluose and bleached using sodium hypochlorite solution. The bleached holocellulose was modified by allylation reaction using allylchloride yielding degree of substitution of 2.33. Graft copolymerization of acrylonitrile onto unmodified and on the allylated derivative was conducted using the potassium-permanganate/toluene redox system in an aqueous medium. The Graft yield for the allylated fiber was generally lower than the value for unmodified fiber, for the latter, graft yield was up-to 80% at 0.63M toluene concentration. The lower graft yield for the allyl fiber was ascribed to the resonance stabilization of the allyl macro radicals. For the allylated fiber, Pg/Pgo is less than unity over the same concentration range of toluene. The lower reactivity of the allyl fiber was attributed to resonance stabilization of the allyl radical species.

Keywords: Plantain Pulp, Acrylonitrile, Copolymer, Allylated fiber, Toluene.

Introduction

Acrylonitrile pertains to a compound which has a general formula CH2CHCN. It is composing of several polymeric substances, including the thermoplastic resins, acrylic textile fibers, synthetic rubbers. Acrylonitrile depicts colorless liquid and has a pungent odor of garlic or onions, mostly used as a solvent in organic synthesis, and as an antiknock agent for gasoline. However, Potassium permanganate, an inorganic compound having a general formula KMnO4 and composed of potassium ion and Manganite(VII)ion. Furthermore, Toluene is an aromatic hydrocarbon with the chemical formula $C_6H_5CH_3$.

Graft polymerization importantly achieves credit within polymer chemistry due to its applicability in the chemical modification of cellulose, used for the directed improvement of these materials (i.e. cloth, fibers, and films). Several methods are aimed at affecting substantial alterations to the chemical and physical properties of polymeric materials. Formation of graft copolymers onto cellulose fibers can be achieved by addition polymerization of vinyl monomers initiated by radicals and ionic species or by condensation polymerization. Generally, graft copolymers are known to be useful in medicine and pharmaceutical industry and with the diversification of the types of hydrophilic monomer grafted onto the fiber. Great time and energy are required for grafting processes and chemical alteration of natural cellulose. In ionic grafting, low molecular weight polymers are obtained whereas free radical grafting can form graft copolymer with high molecular weight polymers. Extracted gum from galactomannans based biopolymers has extensive hydration inadequacy, change in thickness, thereby decreasing their considerable industrial usage [1]. Among the most

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fascinating thermoplastic materials is Acrylonitrile Styrene Acrylate used in an LFAM device as a result of its excellent wettability and mechanical properties [2]. Results showed liquid electrolytes sodium-ion would be replaced by proficient polydactyl linotile-based gel-polymer electrolytes [3]. It was confirmed that the thermal strength of GO-g-PAN reduces through an enhancement in Polyacrylonitrile content bythe thermal gravimetric analysis 4. Grafting of acrylonitrile against cross (alginate/polyvinyl alcohol) CAP beads from thermo-gravimetric study revealed that grafting increases the thermal stability of Cellulose acetate phthalate [5]. In comparing copolymer with nanoparticles counterparts, module of thermo-gravimetric analysis (TGA) showsan enhancement in thermal steadiness, while there is an improved amorphous section of nanoparticles and grafted copolymers by an X-ray diffraction method, and electron microscopy method as well demonstrated [6]. Byway of generalization, the existence of acrylonitrile(AN) monomer, cyclic alkyl amino carbene catalyst bearing phenyl group, and Hoveyda–Grubbs catalysts are less stable than the ruthenium-complex catalyst [7]. A polymer byproduct and potential Geno toxicant, styrene-acrylonitrile (SAN) trimer, was one of the compounds identified in both GC/MS and LC/MS at quantities ranging from 19 to 270 μg g−1. The extractable profile was affected by the filament material, printing procedure, and post-processing method [8]. Permanganate oxidation did not mineralize dissolved organic matter, alkylbenzenes and aromatic compounds composed of nitrogen were treated by $KMnO_4$ to form aliphatic compound containing-nitrogen [9]. Potassium permanganate with ethylene absorbent gives a resourceful manner to search ethylene with an enriched zeolite, which shows areduction in the quality parameters[10].ThepH of potassium permanganate solution was used to oxidized Cotton yarn, oxidized sample; compared with raw cotton, show lesser tensile force, increased wetness sorption, and exterior coarseness [11]. $KMnO_4$strengthen their action and purpose of H2O incorporation, and $KMnO_4$ significance for the machine-driven properties of Potassium permanganatepreserved plant fiberand water-resistant properties [12]. $KMnO_4$ exposure reduced the rate of oxygen evolution and cell growth. This work showed that a high concentration of $KMnO_4$ (10 mg L−1) decreased the fast phase and increased the slow step of QA− re-oxidation kinetics. Electron transport after QA wasblocked, resulting in a considerable amount of QA−re-oxidation being performed via S2(QAQB)−charge recombination [13]. Significantly, Air pre-oxidation (APO) behavior enhanced the structural uniqueness of bio-char, thereby improving its adsorption properties [14]. Experimental evidence demonstrates that gelation of the gels in Canola oil amplifiedthroughflush incorporation towards a concentrated percentage of hexane, as well as known figure of toluene, original solidify gel consisted of drops of water chemically bonded together, more significant immersed volumes results to reduction in gelation of the gel [15]. The utilization of photo-catalytic paints is considered to be the existing method to have toluene reduced, and a noticed compound in this regards for instance polymeric-acrylic, inside coat were identified by gas chromatography–mass spectrometry [16]. The complex oxide of Molybdenum-manganese was used to determine the oxidation of toluene, the selectivity of benzaldehyde and exchange of toluene were rapidly improved by water [17].

The $KMnO_4$ and $C_7H_6O_3$ treated fruits, total chlorophyll incorporation, amount of ascorbic acid reduces and total dissolved solids, content of reducing sugar improved slowly than control fruits during storage [18]. The $KMnO_4$ pre-oxidation enhances the total of algae cells, and this occurrence is vital with the existence of hardness causing ion [19]. Low-exposure to toluene affect the nervous system.Special effects were reversible even at very high contact levels for a very long period. The health effects include lethality, growth, morbidity, liver [20]. Significantly, combined exposure of toluene and xylene are weaker than the effect of contact to the xylene only, and exposure effect is seen higher to toluene [21]. Increasing the potassium permanganate dose lower the negativity in pulp electrolyte leakage, and fresh fruit matter, preserved under a modified atmosphere [22]. Treating $KMnO_4$ withthe disintegration of $C_{14}H_{10}$, and the pyrene onto the soil spike at adiverse concentration significantly reduced Pulmonary arterial hypertension concentration, pyrene($C_{16}H_{10}$) was recalcitrant than phenanthrene [23]. Conventionally, sand filter effluent pretreatment in combined ultra-filtrationprocesses, Presence of potassium-permanganate significantly improves the effectiveness of organic matter exclusion throughout the potassium-permanganate ultra-filtration method [24].Plantain pulp, (Musa spp) as an essential plant fiber source, originated in the region between Malaysia and northern Australia. It is found in all tropical areas where plantain is grown. The baste fibers are cellulosic materials. Cellulose occurs in the form of fiber in a wide variety of growing plants. Graft

Copolymerization of the vinyl monomer is a fascinating method of improving the properties of natural fiber in order to lengthen their application. Consequently, while significant work has been carriedout significantly on grafting of vinyl monomer, particularly acrylonitrile onto various plant fibers, much less has been described on the grafting of acrylonitrile onto alkylated plantain pulp.An examination of the recurrent studies is done and explicitly presented the main conclusion. Potassium permanganate ($KMnO_4$) is useful as a dynamic food-packaging tech performed to be an essential alternative to protect the value, and the protection of freshagricultural harvest to eliminate the C_2H_4 after the nearby atmosphere within the package [25]. At the neutral pH, degeneration of triclosan by potassium-permanganate tracked understandable-first-directive kinetic in a defended deionized water-system, showing the catalytic character of the MnO_2 bent the fit below slightly acidic environment [26]. Permanganate as a useful oxidant for dense non-aqueous phase liquid, rapid pollutant annihilation was express in catalyzed-hydrogen-peroxide schemes, grander pollutant mutilation stayed perceived in permanganate methods. Therefore, the carbon-based substance may consume some given complex connections that resolve influence corrosion efficiency and effectiveness [27]. Noticeably, L-mimosine, been a useful component in propagation and immune reaction, acts as an aide-de-camp in irrefutable immune-suppressive psychoanalysis which reduces the number ofprotractedtissues of agranuloma that are induced by the permanganates of potassium solutions [28]. In natural filtration systems, it was further depicted that Potassium-permanganate, in addition to activated carbon, is less effective, and more appropriate for Trihalo-methanes (THM) reduction strategies conventional water treatment systems [29]. Different $KMnO_4$ concentrations (0.033%-0.066%), on the ductile possessions of cellulose preserved with a 6% sodium hydroxide, matrix adhesion of thermoplastic-polyurethane and composites enhance the NaOH treatment process to strengthen maleic anhydride [30]. Exposure of larvae and young juveniles to $KMnO_4$ in fluctuating salinities revealed that potassium-permanganate is deadly in additional waters to banded low-pitched creepy-crawlies and fingerlings at suggested conduct heights [31]. Potassium-permanganate, sodium perborate rehydrated, and potassium dichromate treated with jute fibers, the maximum inter-facial sticking together through recovering compatibility with poly-propylene were obtained after sodium perborate rehydrated behavior thru given that the utmost superficial coarseness quality of jute [32]. The oxidation of a number aliphatic, aryl aliphatic, and cyclic ketone by potassium permanganate in theacidic medium [33].Toluene, an aromatic organic compound useful in estimating the catalytic enactment of the artifacts as a result of volatility to determine the consequence of the Mn, together with the Co loads on the textures, operational, and the redox possessions [34]. Itis important to note that the Toluene-degrader-lineages established on specific genes were valuable vogueish an exclusive fine-scale penetration bisect of residues occupied at the higher peripheral of the hydrocarbon-plume aimed at comparative discovery, and the quantification examines for aerobics, and the anaerobic redox [35]. The reduction of toluene is achieved by the toting fumigating the methyl, fabricating, andthe catalyzing through the enzymes called benzyl-succinate synthase as well discussed [36]. Incomplete oxidation of toluene,utilizing$VO(acac)_2$ as catalyst precursor, the hydroxyl radical is established to be a dynamic species with an emphasis on the detection of intermediate groups [37]. Predominantly, para-chlorotoluene and ortho-chlorotoluene are generated due to toluene chlorination by electrophilic substitution in acetic acid [38].Generally, water harms on catalytic-oxidation of toluene terminated majority of the Vanadium(V) oxide, enhancing the action and the discernment aimed at benzoic acids. However, reduced temperatures in deactivations by the product of addition are hindered by waters in accumulation [39]. In the existence of oxygen and ultra-violet light, the corrosion of toluene is catalytic, producing benzaldehyde and benzyl alcohol as the products [40]. An acquaintance of N-Europaea to toluene and benzene depict that N-Europaea is a significant chemo-lithoautotroph, trap its energy for development only from the oxidation of ammonia to nitrite [41, 42].

At low temperature, curing increases the development of identical materials thru reduced fracture resistance, the portion of precipitated point, resolute on micro-charts, be situated sophisticated compared to the quantity of other neoprene [43]. Experimentally, infrared spectroscopy is an essentialtool used through the polymer. The rubber scientist for the taxonomy of polymer–amalgam proportion calculations, unrefined material estimations, peaks of nitrite rubber were interrelated by acrylonitrile, precisely by the Kjeldahl technique [44]. The dimerization of acrylonitrile on an efficient catalytic system shows that carboxylic acid is better than hydrogen, as it allows discriminating dimerization of the acrylonitrile's linear dimers with no establishment of propionitrile and the propionitrile is considered to be byproduct as it was demonstrated [45].

There is a reduction in miscibility employing carboxyl terminated acrylonitrile-butadiene containing less acrylonitrile as a result of a change in solubility, whereas epoxy-monomer result to increase miscibility by the copolymer effect [46]. The implantation experiment that was done precisely and explicitly copolymerized by monomers that demonstrated the slightest convoyed by an enriched yield, and the optimistic high-temperature confidence in the 30°C–40°C and a harmful one at upper temperature [47].

Lastly, the experimental graft model that was done further in aqueous display low graft yields, illustrates the attributes for acrylic acid, as less similar to the holo-cellulose sawdust, was experimented [48,49]. The DMAEMA model further on the grafting using the copolymerization-reactions divulge that the munches the DMAEMA monomer speed-up the preliminary percentage of the grafting-copolymerization, although the grafting limitations moderates considerably through accumulative the close of the DMAEMA [50].

Result and Discussion

Within this section, a summarized discussion is presented. Chemical modification of plantain pulp (Musa spp) by allylation reaction yielded the allyl fiber having degree of substitution (D.S) of 2.33 as illustrated in Table **1**.

Table 1: A combined Blank and sample Titration.

VOLUME	RESULT OF BLANK TITRATION (V_o)				RESULT OF SAMPLE TITRATION (V_1)			
	Rough	1st titre	2nd titre	3rd titre	Rough	1st titre	2nd titre	3rd titre
Final Volume (cm^3)	47.10	46.90	46.70	46.80	28.80	28.60	28.40	28.50
Initial Volume (cm^3)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Volume of Thiosulphate used (cm^3)	47.10	46.90	46.70	46.80	28.80	28.60	28.40	28.50

(H+) = 0.49M; (AcoH) = 0.57; fiber = 0.1g; ($KMnO_4$) =8.33X10-3M; POT =10min (AN)= 0.25M; Temperature = 300C; Time= 3h.For the unmodified fiber, graft yield is in the range of 50-80% in the presence of toluene in the concentration range of 0.15-0.63M. In the presence of toluene, the graft yield decreased and then increased over the concentration range 0.15-0.63M.Graft yield is enhanced by the presence of toluene having Pg/Pg0 of up to 8.0 at 0.15 and 0.63M concentration of toluene for the graft copolymerization reaction using redox pair, $KMnO_4$, and toluene, the initiation species, for the graft reaction, respectively.

Since Pg/Po is greater than 1 in the presence of toluene, than the benzyl radical species (I) is an effective initiator of the graft reaction. This shows much lower levels of graft yield i.e. grafting onto allylated fiber gave yield in the range of 20-30 in the presence of toluene in the concentration range 0.15-0.63M as depicted in Table **3**. At the corresponding concentration of toluene, graft yield for the unmodified fiber are higher by up to 5-fold than the value for the allylated fiber. For initiation of allylated fiber, the initiated species and the initiated allyl cellulose can undergo resonance stabilization respectively.

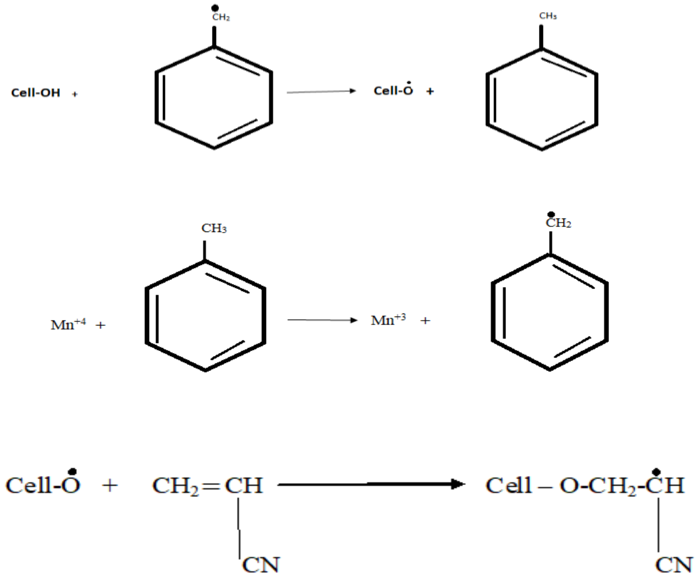
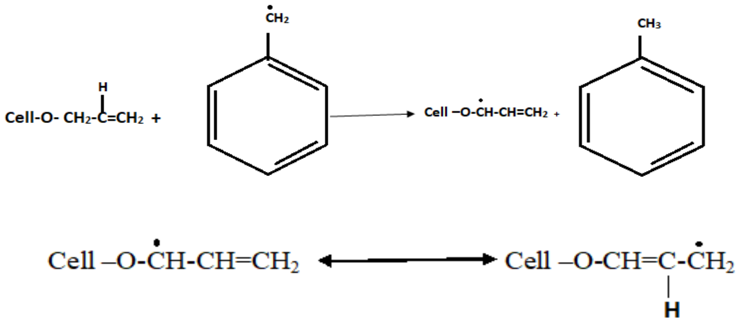


Table 2: Effect of Toluene On Grafting Of Acrylonitrile Onto Unmodified Plantain Fiber.

S/N	TOLUENE(M)	GRAFT YIELD(%)	Pg/Pg_0
1	0	10	
2	0.15	80	8.0
3	0.31	70	7.0
4	0.47	50	5.0
5	0.63	80	8.0

Table 3: Effect of Toluene on Grafting of Acrylonitrile onto Allylated Plantain Fiber.

S/N	TOLUENE(M)	GRAFT YIELD(%)	Pg/Pg_0
1	0	60	
2	0.15	20	0.33
3	0.31	20	0.33
4	0.47	10	0.17
5	0.63	30	0.5



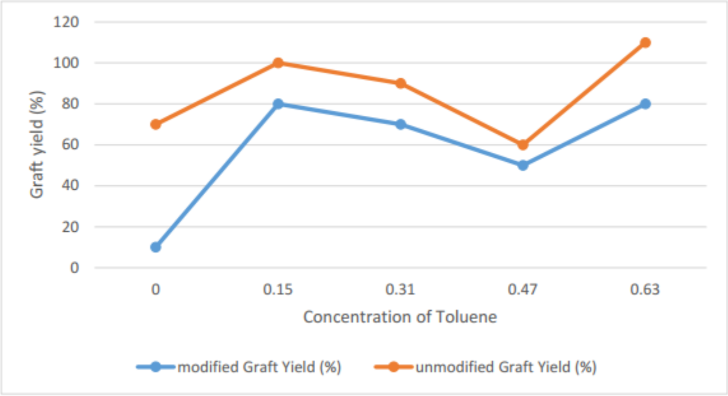


Figure 1. Effect of Toluene on Graft yield of Modified and unmodified Sample.

The lower reactivity of the resonance stabilized allyl cellulose radicals account for the observed lower graft yield.It is noted however that for allyl cellulose Pg/Po is lower than the presence of toluene was minimal to the graft yield that is to say abstraction of hydrogen from the allyl cellulose by benzyl radicals is less effective than abstraction of hydrogen from the unmodified cellulose as figure 1 illustrates.

Conclusion

The Potassium permanganate/toluene redox system can initiate the graft copolymerization of acrylonitrile onto plantain pulp fiber and its allylated derivative in an aqueous medium at 30^{0}C.Allylation of plantain pulp fiber gave value of 2.33 for degree of substitution and this suggested that two hydroxyl groups of the anhydroglucose units were substituted. Graft yield for the allylatedfiber were generally lower than those for the unmodified fiber by as much as 70% at 8.33 X 10-3M concentration of permanganate. This was ascribed to resonance stabilization of allylated radical species, which rendered them less reactive. The ratio Pg/Pgo which is the graft yields of toluene correspondingly is superior than 1 at the toluene concentration 0.15 – 0.63M for the unmodified fiber. For the allylatedfiber, Pg/Pgo is less than 1 over the same concentration of toluene.

Experimental Section

In this section, we present the method and the numerical results are examined, briefed experiment is as follows. A 5% NaOH solution was added to the acrylonitrile monomer in a separating funnel and the mixture was shaked for 20-30 minutes. The aqueous layer was decanted and the monomer was washed with 5% NaCl solution. The monomer dried up in excess anhydrous calcium chloride, then distilled fractionally. Toluene was treated with sulphuricacid solution, then eroded several times with distilled waters to a neutral pH using litmus paper and then dehydrated in excess of anhydrous CaCl overnight; the toluene was then fractionally distilled. The plantain pulp attained from the University premises of FederalUniversity Agriculture Abeokuta. The bastfibers were midair-withered together subject to flattened in a plaster to soften and increase its surface area. It was refluxed for one hour to remove waxes and resinous materials. The holocellulose were got thoroughly sweep absent with disinfected waters, and then it was exposed to air for drying.Bleaching of holocelulose was done with sodium hypochlorite bleaching agent. It was washed with excess purified waters to unbiased the pH and then exposed to air for drying. Reaction for the manufacture of bleaching agent can be exemplified as tail.

$$2KMnO_4 + 16HCl2 \rightarrow MnCl_{2(aq)} + 2KCl_{(aq)} + 8H_2O + 5Cl_{2(g)}$$

$$2NaOH + Cl_2 \longrightarrow NaOCl_{(aq)} + NaCl_{(aq)} + H_2O_{(l)}$$

Allylation of the plantain fiber was done by reacting it with aqueous sodium hydroxide. The fiber was then refluxed with solution of allyl chloride, the fiber was rinsed diethylether and air dried after refluxing. Summary of the reaction for the allylation of the plantain fiber is as follow:

$$NaOHCH_2{=}CHCH_2Cl + Cell\text{-}OHCell\text{-}ONa + H_2O \longrightarrow Cell\text{-}OCH_2CH{=}CH_2 + NaCl$$

The formula below was used to calculate the number of mole(s) of allyl content of the fiber:

Determination of Degree of Allylation

The amount of allyl moiety in the fiber was determined by reacting 0.5g of allylfiber with 25ml of 0.1M iodine solution for one hour. Then back titrate the unreacted iodine with 0.05M thiosulphate solution and the reaction was carried out three times. The color changes from brick red to black then to colorless. 1% starch solution was used as an indicator.

$$\frac{(Vo - V1).Mt.162}{Ws.127}$$

Given that N depicts a number of mole(s) of allyl moiety given eachthe AGU of the fiber, Vo shows volume of blank solution, V1depicts the volume of sample solution, Mtillustrates Molarity of thiosulphate equaling to 0.05,Ws portrays Weight of the allylfiber.0.1g of the modified fiber was immersed in a measured quantity of distilled water in the reaction vessel. Measured quantities of glacial acetic acid, concentrated sulphuric acid solution, toluene, potassium permanganate was introduced into the reaction vessel, such that the total volume of the reaction was made up to approximately 30mls.

An initiation time of ten minutes was allowed that is to say time for the potassium permanganate and toluene redox pair initiator to react with the fiber, before the introduction of a measured quantity of acrylonitrile monomer. The reaction was allowed to proceed for three hours before it was then terminated with 50ml of distilled water. The grafted polymer was then sluicedthrough distilled-waters, and then exposed to desiccated in the hotter at 40^{o}C overnight. The total weight obtained minus the weight of the sintered glass gave the weight of the grafted polymer.

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