Synthesis and Functional Utilities of Starch Graft Copolymerized Products, Corn Starch and Tapioca Starch Derived Biomaterials

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Abstract

Starch is a vital food product and a versatile biomaterial employed across various industries, including food, health, textiles, chemicals, and engineering globally. Its adaptability for industrial applications is primarily determined by its functionality and physicochemical properties. Advances in chemistry and biotechnology have led to numerous modifications of starch for diverse uses beyond its natural state. To enhance the biocompatibility, biodegradability, and physical characteristics of starch-based biodegradable polymers, various strategies are essential. This study explores the synthesis and functional applications of starch graft copolymer products derived from corn and tapioca starch. It specifically examines the synthetic techniques and utility assessments of several starch-based grafted materials, including maleic anhydride copolymers, substituted amoxicillin grafts, PMMA-grafted starch copolymers, and modifications of corn and tapioca starch through cationization and sulfonation, among others. The novelty in the current studies lies in the transition of these materials from basic biodegradable fillers to high-performance, stimuli-responsive biomaterials tailored for advanced industrial and medical applications. The key advancements focus on synergistic blending, "green" rapid synthesis, and the development of active, intelligent properties that native starch lacks. The work comprises a review of green synthetic approaches and functional utility assessments of synergistic multi-starch formulations.

Keywords: Biomaterials; Corn starch; Functionalization; Graft co-polymerization; Tapioca starch

1. Introduction

Starch, a plant-based carbohydrate polymer, is a staple food material. The extensive use of starch materials in food and non-food industries is attributed to their high abundance, non-toxicity, low cost, renewability, biocompatibility and biodegradability. (Bušić et al., 2018) Starch in its native form has been modified by different methods over the past decades to get the starch functionalized materials of various industrial applications. (Salimi et al., 2023) The novel, environment friendly and efficient methods of starch modification are being developed (Sun et al., 2023) and these modifications are associated with structural changes so as to bring about the changes in functional properties. The microstructure of starch greatly determines the modification techniques to be used and the digestibility of modified starch materials. (Lu et al., 2023; Jiang et al., 2023) The current study has incorporated the syntheses and functional utility aspects of starch graft co-polymerized products, corn starch and tapioca starch derived biomaterials. In particular, the study focuses on step-wise synthetic techniques and utility assessments of the graft co-polymerized starch, maleic anhydride and substituted amoxicillin drug copolymer grafted starch materials, starch grafted methyl nadic anhydride and substituted 4-amino-antipyrine, poly(methyl methacrylate) (PMMA) -grafted starch copolymers, cationic starch-grafted-cationic polyacrylamide (CS-g-CPAM), hydroxypropyl sulfonated and caproylated corn starch (HCS), and quaternization/hydroxypropyl sulfonation of oxidized tapioca starch (OTS) materials. While the current review establishes the state of starch graft copolymers, the research gap and industrial problems are mainly associated with the inverse relationship between grafting percentage (GP) and grafting efficiency (GE). Increasing the concentration of monomers leads to more grafts (high GP), but it also triggers the formation of unwanted homopolymers (low GE). It may not be plausible to achieve the copolymers with both high GP and GE simultaneously without complex, multi-step additions, that are difficult to replicate at an actual industrial mass production from a laboratory based green syntheses.

2. Graft-Copolymerization of Starch: Macroinitiator based Starch Activation

Graft-copolymerization of starch can be obtained by two steps of reaction. In the first step, starch is activated to produce a macroinitiator, and in the second step, there is graft-copolymerization of polymethyl methacrylate onto the macroinitiator (Fig. 1). Macroinitiator steps can be performed *via* the addition of an acyl bromide compound solution in the presence of catalyst triethylamine (TEA), pyridine (Py) and 2,2-bipyridyl (Bpy). The starch activation product prior to the synthesis of the macroinitiator is greatly influenced by temperature and the type of catalyst used. According to Mohamad, pyridine becomes the best catalyst which gives the highest degree of starch substitution. (Mochamad et al., 2014) The starch graft copolymers are widely used in industries as flocculants, films, super absorbents and oil drilling agents. They are used for removal of heavy metal ions in waste water treatment processes. They are used as additive to increase the biodegradability of polymers. (Jyothi, 2010) Based on amylopectin from native waxy starches, surfactant-free starch-graft-copolymer latexes can be produced with little degradation if the precursor particles are stabilized against coalescence early in the reaction. This method may have the advantage that most of the starch is grafted onto synthetic latex particles, which prevents it from migrating from the hydrophobic layer that forms on the substrate surface in wet applications like coatings for paper. (de Bruyn et al., 2007)

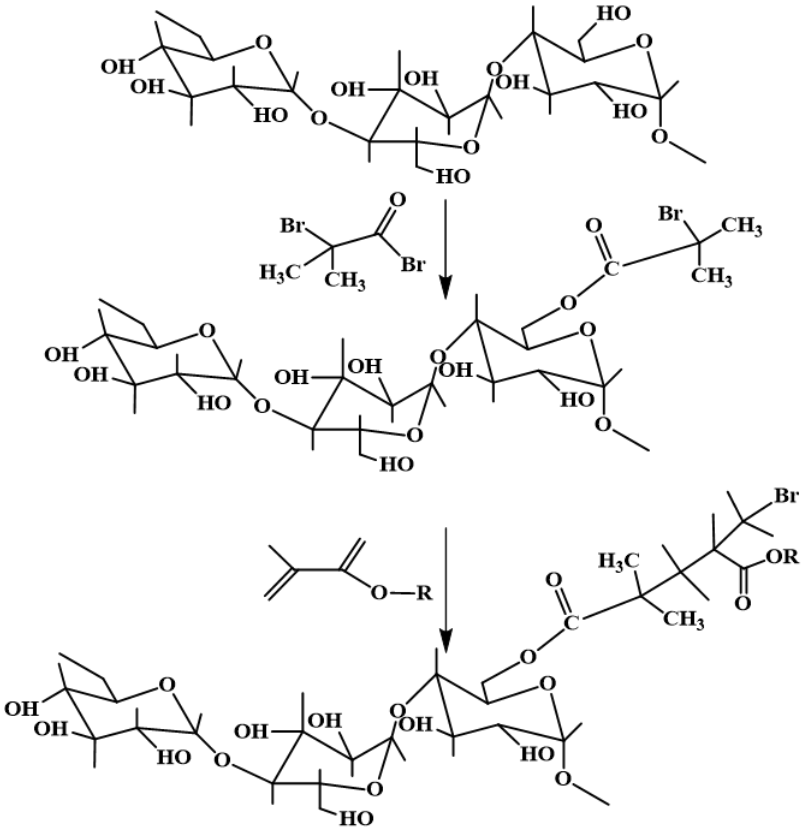

Fig. 1: Graft-copolymerization of starch

3. Starch Grafted Maleic Anhydride and Substituted Amoxicillin Drug Copolymer: Biodegradable Pro-Drug Carrier

Graft polymerization of starch can be initiated by using many initiators like $KMnO_4$/organic acid, redox pair Fe^{2+}/H_2O_2, potassium persulfate, transition metal ions/organic reactors. The ceric ions, Ce(IV) have been found to be interesting initiators due to very low homopolymer formation and high graft yield, and the ceric ions are redox initiators with high grafting efficiency. (Nguyen & Nguyen, 2010) The ceric salts like cerium ammonium nitrate (CAN) can be used as initiators for the grafting of vinyl monomers into glucose, and this process involves the formation of a ceric ion-glucose complex that is further dissociated into cerous, Ce(III) ions (AL-Shihani, 2016) and the generation of glucose radicals on the O-atom *via* the deprotonation of glucose. (Nguyen & Nguyen, 2010; Kalia & Sabaa, 2013) Starch plays a vital role in graft copolymerization due to the presence of hydroxyl groups. When starch is treated with maleic anhydride in the presence of CAN as a radical initiator, a graft copolymer that acts as a biodegradable pro-drug carrier is formed. A new drug polymer has been found to be obtained by the reaction of starch with maleic anhydride and the consequent substitution with amoxicillin. (Alsalami & Farhan, 2017) In this particular modification, maleic anhydride (M2) acts as a spacer, while CAN acts as an initiator (Neelam et al. 2012) and amoxicillin as an amino drug (Fig. 2). (Alsalami & Farhan, 2017) The graft copolymerization of starch with amoxicillin brings about the higher thermal stability of the drug polymer than the drug alone, and it extends the expiry date by protecting the drug stability. (Alsalami & Farhan, 2017) The polymeric nanoparticles' intensity of charge is influenced by the surface of a bacterial biofilm. This stops the biofilm from becoming further enriched with polymeric nanoparticles. The presence of extracellular polymeric substances makes it difficult for poly(ethylene)-glycolylated nanoparticles and other negatively charged nanoparticles to penetrate deeply into the biofilm, despite their ability to have extended blood circulation periods and strong biocompatibility. The polymeric nanoparticles were loaded with antibiotics by the process of self-assembly. The concentrations of antibiotics in these nanoparticles are restricted by this method. Increasing the amounts of antibiotics that these nanoparticles carry into the biofilm's deepest pockets is difficult. Thus, the fields of anti-biofilms could benefit from the application of light, heat, and magnetic fields. (Li et al., 2021)

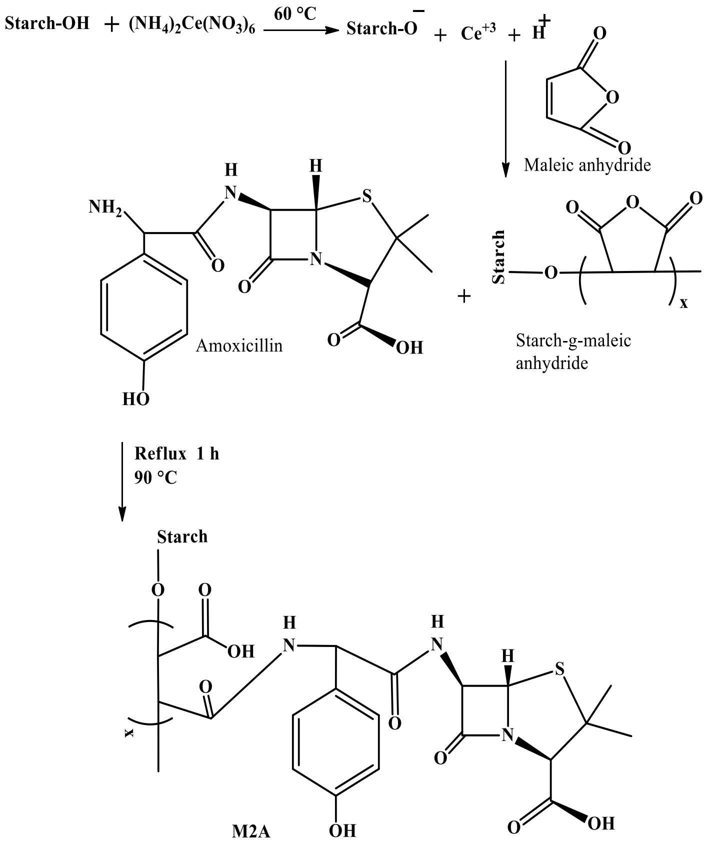

Fig. 2. Starch-g- maleic anhydride and substituted amoxicillin drug copolymer

4. Starch Grafted Methyl Nadic Anhydride and Substituted 4-Amino-Antipyrine: Target Site Delivery Materials

The advent of new properties in tissue engineering and tissue adhesion (Athawale & Lele, 2000; Cha et al., 2000; van Hest & Tirrell, 2001) has introduced the advancement in grafting of starch with acrylic/vinyl monomers such as acrylic acid, acrylamide, acrylonitrile, methacrylic acid, methacryl amide, methacrylo nitrile, and vinyl acetate (Kiatkamjornwong et al., 2002; Singh et al., 2006) The modification of drugs with these starch-based polymers, which can be released in a controlled manner, has been found to increase the concentration of drugs at the target site. These polymers can be used for the generation of biocompatible materials of medical and pharmaceutical applications. (Kalshetti et al., 2012) The release rates of the drugs obtained from biodegradable polymers can be controlled *via* a number of factors like the biodegradation kinetics of the polymers. (Al-Sabagha et al., 2009; Zhu et al., 2008) The 4-amino-antipyrine has been reported to protect against oxidative stress and cancer and various antipyrine derivatives were evaluated as having anti-inflammatory, analgesic, anticancer, and antimicrobial activity. (Deshmukh et al., 2015) For the grafting of vinyl monomers into starch, CAN could be used as an initiator. The complex of ceric ion-starch first formed gets dissociated to give the cerous ions (Ali & Farhan, 2017) and starch radicals are generated by hydrogen abstraction from starch. (Nguyen & Nguyen, 2010; Kalia & Sabaa, 2013) For the graft copolymerization of starch, it has hydroxyl groups that act as active sites. (Ali & Farhan, 2019) The graft of starch with methyl nadic anhydride was found to get substituted by 4-amino antipyrine to give a new drug copolymer (Fig. 3) with the sustained release behavior and minimal side effects (Ali & Farhan, 2019) The NH_2 group of drugs acts as a strong nucleophile, which attacks the carbonyl group (C=O) of methyl nadic anhydride (Fig. 4) to give N-drug substituted copolymer. (Umar et al., 2016)

5. Starch-G-PMMA Copolymer as a Green Target Drug Delivery Material: Free Radical Polymerization Process

The mechanical and physical properties of the Poly(methyl methacrylate) PMMA-grafted starch copolymers are associated with their ability to displace synthetic polymers from their conventional usage. The finished product will benefit customers and be reasonably priced as well. In the near future, the usage of such starch-based polymers will also greatly reduce the environmental damage caused by the use of synthetic polymers. More work is being done by researchers to create materials based on biopolymers in an effort to completely replace synthetic polymers. (Dhar et al., 2023) The modification of starch has been reported to be caried out by grafting with polymethyl methacrylamide (PMMA) in the presence of ammonium persulfate (APS) as an initiator *via* a simple free radical polymerization process (Fig. 5). The copolymer obtained in this way has been found to be applied in the target drug delivery. (Umar et al., 2016)

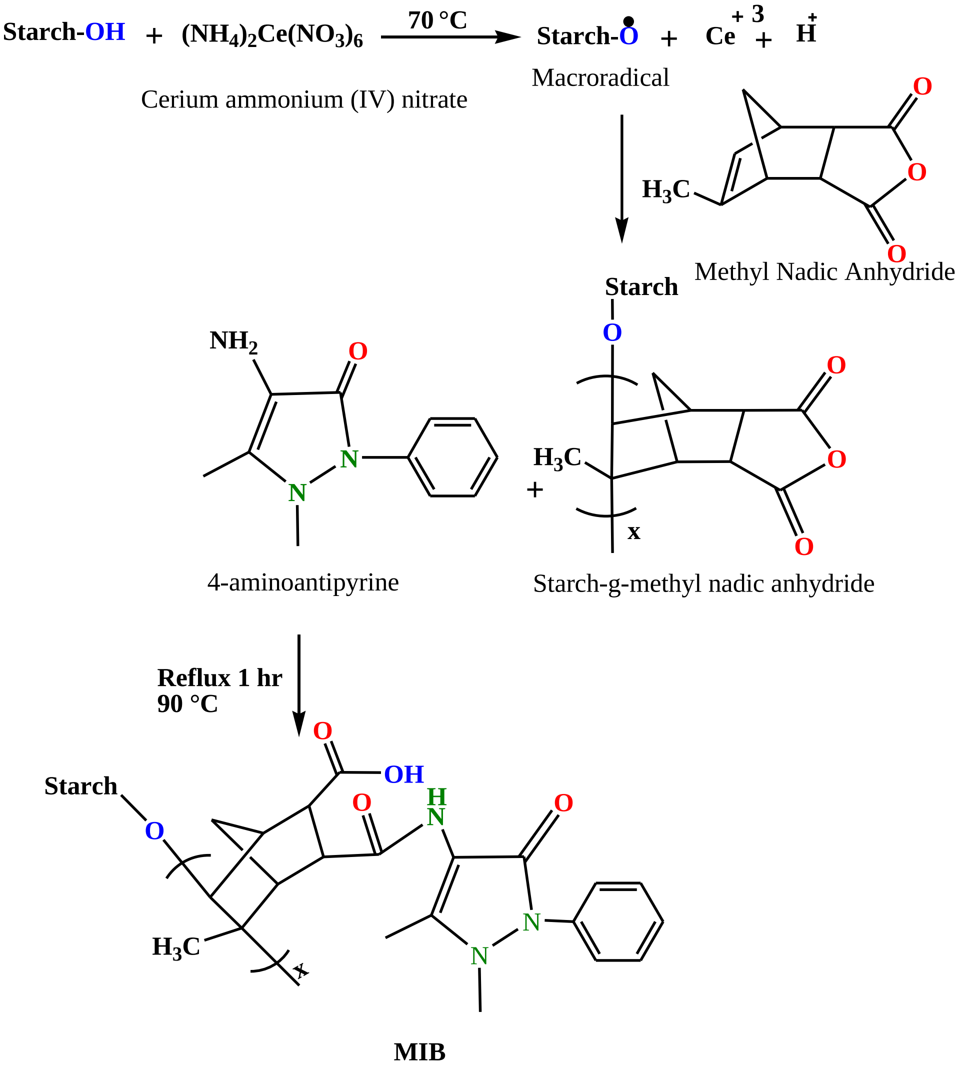

Fig. 3. Starch-g-methyl nadic anhydride and its substitution with 4-amino-antipyrine.

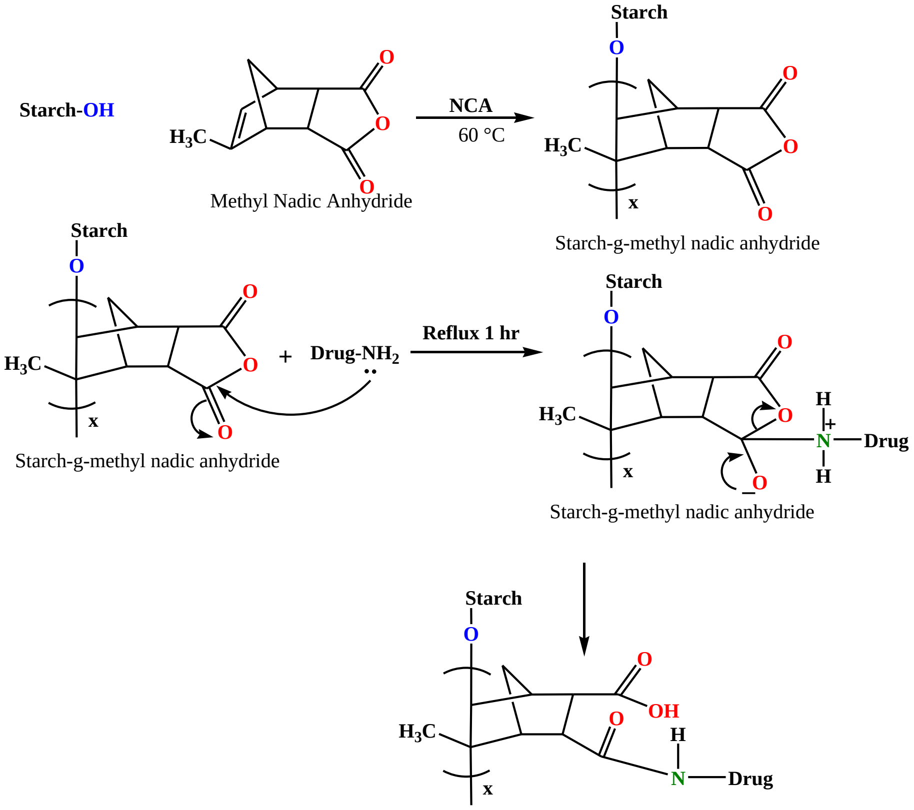

Fig. 4. Mechanism of ring opening reaction of starch-g-methyl nadic anhydride by nucleophilic reaction.

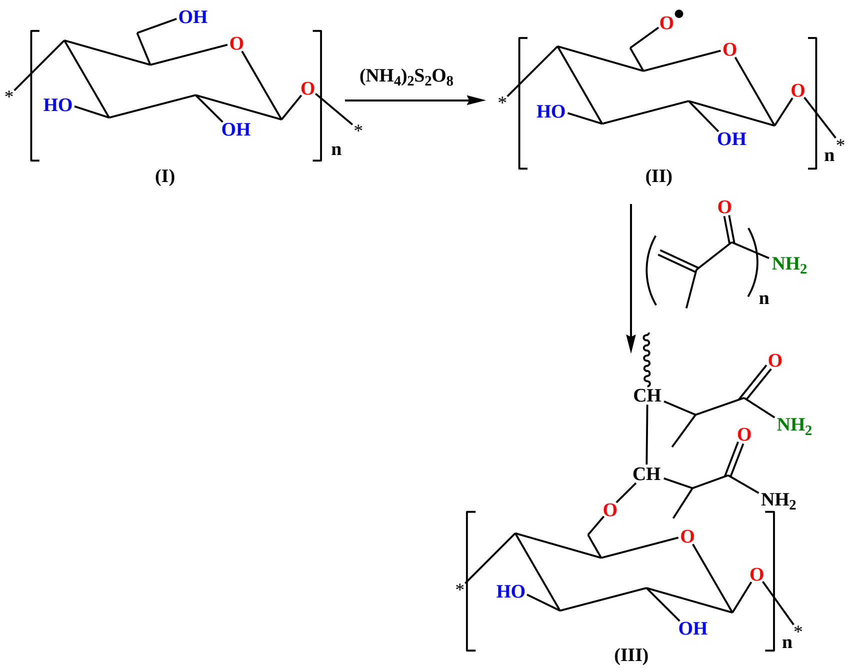

Fig. 5. (I) Starch, (II) starch-macroradicals, and (III) starch-g-PMAA copolymer.

6. Cationic Starch-Grafted-Cationic Polyacrylamide (CS-G-CPAM) as a Novel Flocculant: Graft Polymerization of Free Radicals

The cationic polyacrylamide (CPAM) has been found to have excellent properties of flocculation, turbidity removal, and decolorization. So, oil sludge has been widely used as a treating agent. (Lee et al., 2011) Negatively charged particles of oil sludge, electrical neutralization of CPAM and bridging adsorption with oil sludge particles provide settlement, aggregation and destabilization of the oil sludge particles to get the desired flocculation. At present, extensively and deeply used CPAMs are mainly polyacrylamide-acryloyloxyethyl trimethylammonium chloride P(AM-DAC), polyacrylamide-demithyl diallyl ammonium chloride P(AM-DMDAAC) and polyacrylamide-methacryloxyethyl trimethylammonium chloride P (AM-DMC). Other series of CPAMs are still in the process of experimental research. (Kumar et al., 2015; Huang et al., 2015) The CPAM is extensively used, but it shows limitations of difficulties in biodegradation to cause secondary environmental pollution. (Bolto & Gregory, 2007) and it is disadvantaged by low resistance to alkali. (Ma et al., 2017) These problems can be overcome by the use of biodegradable and ecofriendly derivatives of starch, cellulose, and chitosan. (Song et al., 2013) The cationic degree of starch can be increased by chemical modification to impart the charge neutralization. However, under neutral conditions, the solubility of starch polymer flocculant is weak. (Jia et al., 2016) The CPAM modified by graft copolymerization has been found to be more soluble, more effective flocculant and environment-friendly copolymer. (Sun et al., 2017) The cationic starch-grafted-cationic polyacrylamide **(**CS-g-CPAM) is a novel flocculant which can be synthesized by initiation polymerization of ammonium per sulfate. At first, cationic starch (CS) is obtained from starch (ST) *via* an etherification reaction. Then, under the action of the ammonium per-sulfate initiator, CS, AM, and DMDAAC monomers give free radicals. Finally, CS-g-CPAM flocculant can be obtained by the graft polymerization of free radicals (Fig. 6). (Ying et al., 2019)

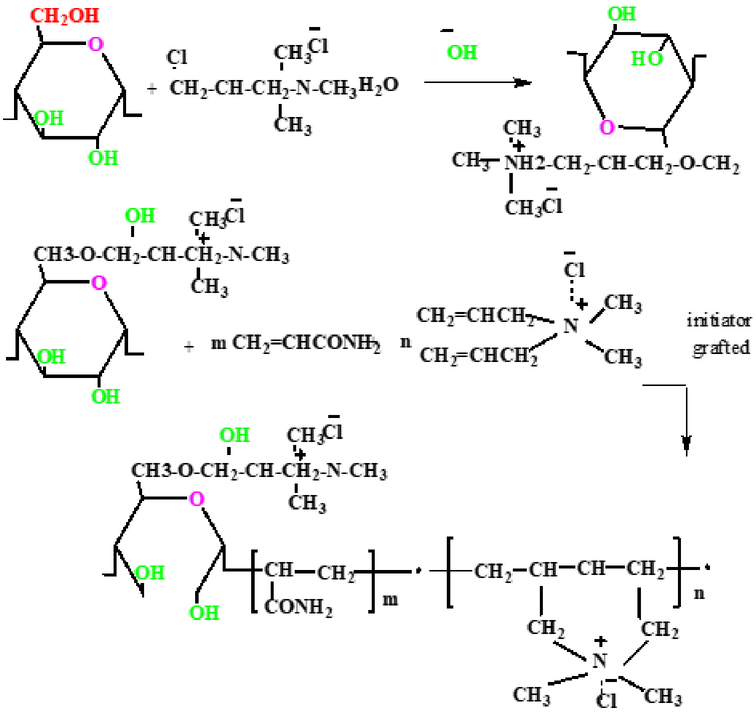

Fig. 6: Reaction mechanism of the synthesis of CS-g-CPAM

7. Hydroxypropyl Sulfonated and Caproylated Corn Starch (HCS): Enhancement in Adhesion and Film Properties

The hydroxypropyl sulfonated and caproylated corn starch (HCS) samples with different degrees of substitution (DS) were synthesized by a hydroxypropylsulfonation of acid-converted corn starch (ACS) with 3-chloro-2-hydroxy-1-propanesulfonic acid sodium salt (CHPS-Na) and subsequently a caproylation with caproic anhydride (CA). The chemical structure of hydroxypropyl sulfonated and caproylated corn starch (HCS) investigated is described in Fig. 7. Hydrophobic caproates that contain ester groups also favor increasing the Van der Waals force at the interfaces between starch adhesive layers and polylactic acid (PLA) fibers. For these reasons, the modification improved the adhesion. Considering the results of HCS in paste stability, adhesion, film properties, and preparation cost, HCS demonstrated potential applications in PLA warp sizing and papermaking. (Li et al., 2019)

Step-1

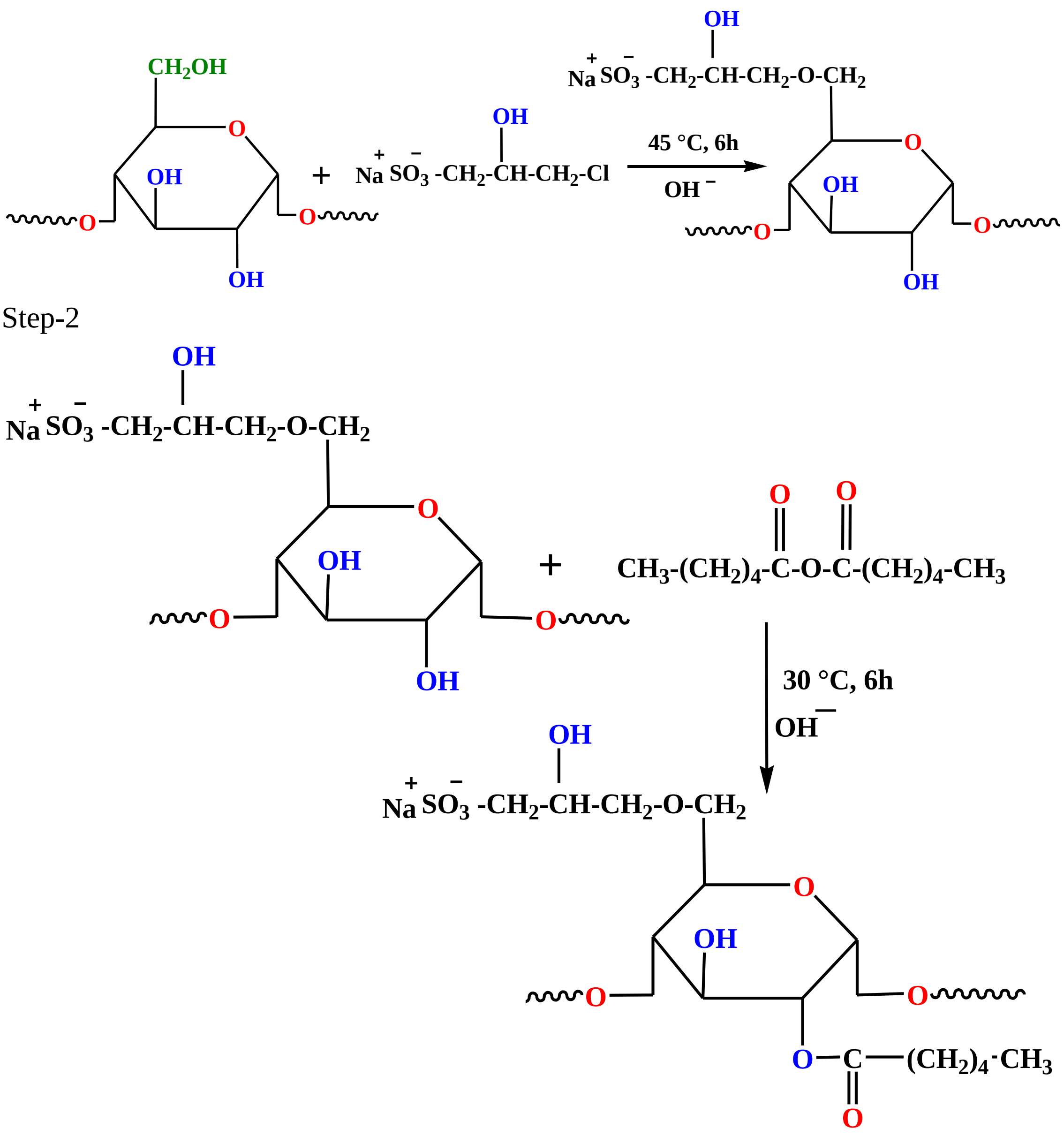

Fig. 7: Hydroxypropyl sulfonation and caproylation of corn starch for the synthesis of HCS.

8. Quaternization/Hydroxypropyl Sulfonation of Oxidized Tapiocal Starch (OTS)

By the simultaneous use of quaternization and hydroxypropyl sulfonation of oxidized tapioca starch (OTS) with N-(3-Chloro-2-hydroxypropyl) trimethylammonium chloride (CHPTAC) and 3-Chloro-2-hydroxy-1-propane sulfonic acid sodium salt (CHPS-Na), a series of quaternization and hydroxypropyl sulfonation of oxidized tapioca starch (OTS) samples can be obtained. (Li et al., 2018) The oxidation of starch with NaClO for preparing OTS (Fig. 8) in step I and quaternization and hydroxypropyl sulfonation of OTS with CHPS-Na (Fig. 9) in step II show the overall process of quaternization and hydroxypropyl sulfonation of oxidized tapioca starch (OTS) for the preparation of quaternized and hydroxypropyl sulfonated OTS *viz.* QHOTS. For sizing yarns made of synthetic and natural fibers or for surface sizing in the production of paper, OTS is used in traditional size formulations. The viscosity of cooked OTS paste tends to decrease throughout the size procedure as a result of the cleavage of D-glucosidic linkages. Thus, OTS paste showed poor stability affecting product quality. (Zhu et al., 2013) The paste viscosity of starch can be stabilized *via* the process of cross-linking of granular starch with succinic acid (Zhu et al., 2013) or formaldehyde. (Zhou, 2004; Zhu & Cao, 2004) The desired effect has been seen with epichlorohydrin as the cross-linking agent. (Hamdi et al., 2008) But serious health problems can be caused by formaldehyde (Li & Yang, 2011) and epichlorohydrin is harmful for human eyes and breathing organs. (Lawrence et al., 1972)

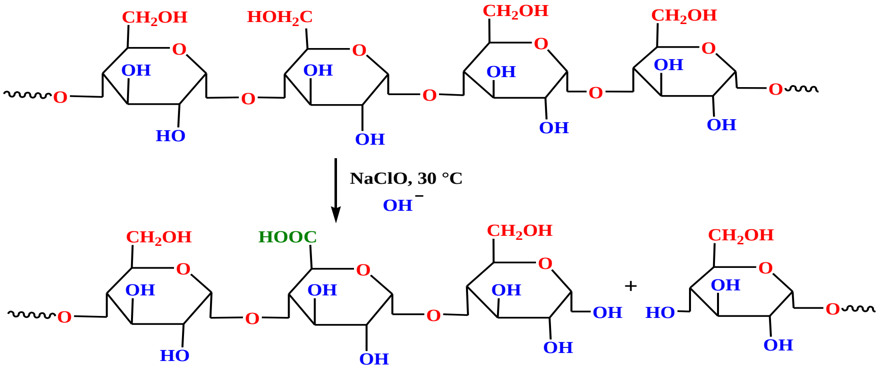

Fig. 8: Quaternization and hydroxypropyl sulfonation of OTS for the preparation of QHOTS: Step I: oxidation of starch with NaClO to give OTS

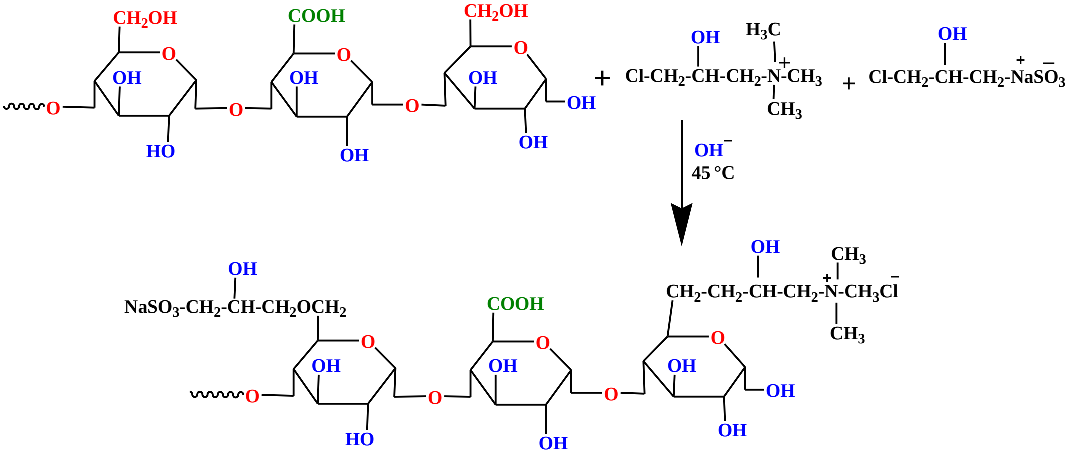

Fig. 9: Quaternization and hydroxypropyl sulfonation of OTS for the preparation of QHOTS: Step II: quaternization and hydroxypropyl sulfonation of OTS with CHPS-Na

The functional and inherent structural properties of starch are essential and valuable for sizing in textile and papermaking industries. (Zhu & Cheng, 2008) The two-step technique can be applied to introduce anionic and cationic groups into starch molecules. (Li & Zhu, 2016) The simultaneous introduction of positively and negatively charged starch derivatives makes the solution overcome the insufficiencies of anionic and cationic starch polymers. The functionalization of starch by one-step method as quaternization and hydroxy propyl sulfonation involves 3-(trimethylammonium chloride) - 2-hydroxypropyl (TMACHP) as cationic and 3-propane sulfonic-2-hydroxy (PSH) as anionic groups. (Li et al., 2018) The introduction of TMACHP and PSH groups in OTS molecules improves the adhesion, reduces the brittleness of film and increases the viscosity (Li et al., 2018). This process of introduction of TMACHP and PSH groups in OTS molecules has also been reported to increase the paste flow resistance. (Zhu & Wang, 2014)

9. Conclusion

The versatility and sustainability of starch as a fundamental carbohydrate polymer are underscored by ongoing advancements in its modification techniques, which enhance its functional properties for diverse industrial applications. This study highlights the innovative synthesis and utility of various graft co-polymerized starch materials, paving the way for future developments in both food and non-food sector applications. Starch as a multifunctional biomaterial lends itself to modifications with innovative synthetic techniques so as to obtain the starch functionalized derivatives of structural alterations. By exploring various modifications of corn and tapioca starch, the current study shows the potential of starch graft copolymers in advancing biocompatible and biodegradable solutions across the multiple sectors of food and non-food industries and pharmaceutical applications. The studies on corn and tapioca starch-derived biomaterials provide unique advancements by moving beyond simple starch modification to create high-performance, functionalized green composites and targeted medical delivery systems. Their unique contribution lies in developing stimuli-responsive systems that allow for "smart" food packaging and the precise, pH-dependent release of covalently bonded drugs.

Conflicts of Interest

The authors have no conflicts of interest to declare.

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