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Synthesis and Research of Poly [methyl-3-(*tetraacetyl mannoside*) propyl] siloxane for use in industrial production

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Abstract

Biopolymer products have secured an important position in industrial technology due to their unique properties, including thermal stability, flexibility, and resistance to environmental factors. This article discusses the synthesis and characterization of Poly [methyl-3- (*tetraacetyl mannoside*) propyl] siloxane (PMMaPS). The physicochemical characteristics and elemental analysis of the synthesized product were studied by FTIR, ^{1}H NMR, ^{13}C NMR spectrophotometer and scanning electron microscope (SEM).

Keywords: synthesis, polymers, PMMaPS, PMHS, siloxane

Introduction

Natural and synthetic polymers due to its non-toxicity, biocompatibility, biodegradebility, chemical reactivity, and microbe resistance, it has excellent potential for various scientific uses in biomedical, food, agricultural, pharmaceutical, and other industries. The many advances in glycoscience have more and more brought to light the crucial role of glycosides and glycoconjugates in biological processes [1-4].

Additional interest in glycoside synthesis has arisen with the prospect of producing sustainable materials from these abundant polymers **[5-7].** Among the bio catalytic strategies, glycosynthases, genetically engineered glycosides void of hydrolytic activity, have gained much interest in recent years, enabling not only the selective synthesis of small glycosides and glycoconjugates, but also the production of highly functionalized polysaccharides.

Modified mono- and polysaccharide derivatives play an important role in discovering new sustainable materials as they originate from renewable resources. Polymers are defined as thermoplastic and thermoset polymers and elastomers. Polymers which are biodegradable currently achieve high interest in materials science. Specific initiators and catalysts are used to initiate and accelerate the chemical reactions between monomers **[8].** Poly[methyl-3-(tetraacetylmannoside) propyl] siloxane (PMMaPS) was synthesized by acetylation of mannopyranoside, allylation of the resulting product, and subsequent hydrosilylation using our selected chosen catalysts.

Materials and Methods

Starting materials and reagents required for the synthesis Polymethyl mannoside polysiloxane (PMMaPS): 1,2,3,4,6-penta-O-acetyl-β-D-mannopyranose, allyl alcohol; [(BF_3•$O(CH)_2$)] - for allylation as a catalyst; toluene, methanol; Pt/C - for silylation catalyst and poly(methylhydro)-siloxane (PMHS), were purchased from Aldrich and used without further purification. The Chemical structure and elemental analysis of the synthesized product PMMaPS were studied by FTIR, ^{1}H NMR, ^{13}C NMR spectrophotometer and scanning electron microscope (SEM).

Fourier transform infrared (FTIR) *spectra of the samples were recorded using a Shimadzu IR Affinity-1S FTIR spectrophotometer. The spectra were collected in the range of 4000-700 cm $^{-1}$ with 32 scans at a resolution of 4 cm $^{-1}$.*

^{13}C NMR and ^{1}H NMR spectra *of the compound were recorded on a 400 MHz spectrometer using $CDCl_3$ as the solvent.*

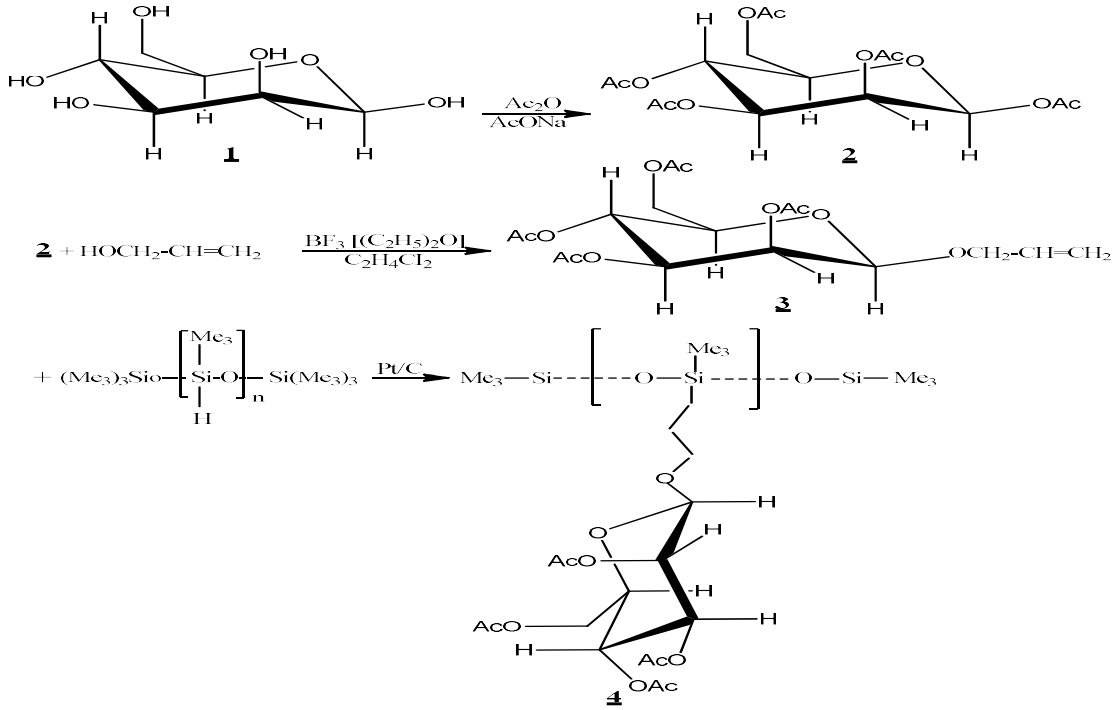
Scanning electron microscope (SEM) *of the synthesized product (PMMaPS). The surface morphology and elemental composition of the samples were examined using a ZEISS Sigma 300 scanning electron microscope. The maximum accelerating voltage was 30 kV, and the spatial resolution was 2.0 nm. The microscope was equipped with a water-cooled lens system that maintained the operating temperature at 19.5 °C, ensuring a stable magnetic field during analysis.*

Results and Discussion

Synthesis of allyl-2,3,4,6-Tetra-O-acetyl-β-D-mannopyranose (3) [9]. To a solution of 2.34 g of β-pentaacetate and 3 ml of allyl alcohol in 44 ml of CH_2Cl_2 add 8 ml of BF_3•Et_2O, stir for 5.5 h at 0^0C. Dilute with ethyl acetate and wash with $NaHCO_3$. Leave the filtrate overnight over Na_2SO_4. Concentrate the solution under vacuum. A colored syrup is obtained. The product crystallizes from a mixture of ethanol and hexane. Yield 1.4 g (60.3%). $T_{melt.}$ 85 ^{0}C.

Synthesis of poly[methyl-3-(*tetraacetyl mannoside*) *propyl*]siloxane (4). 1.12 g of poly(methyl-hydro)siloxane were placed in a reaction flask and 1.4 g of allyl-2,3,4,6-tetra-O-acetyl-β-D-

mannopyranose were added. The reaction was carried out under nitrogen atmosphere. 3-4 drops of platinum Pt/C catalyst were added to 20 ml of freshly distilled toluene. The reaction solution was heated to 75 ^{0}C with constant stirring. After 65 minutes, 7 ml of methanol was added dropwise with vigorous stirring. The precipitated polymer was dissolved in toluene and freeze-dried. 2.1 g (106.0.%) of poly[methyl-3-(*tetraacetyl mannoside*)propyl]siloxane are obtained. The melting point was 273 °C. The chemical structure, composition, and elemental characteristics of compound **4** were investigated using FTIR spectroscopy, ^{1}H NMR and ^{13}C NMR spectrometry, as well as scanning electron microscopy (SEM).



Scheme 1. Synthesis of PMMaPS

FTIR spectrum of the PMMaPS. In the FTIR spectrum exhibits a strong ester carbonyl absorption band at 1742 cm^{-1}, which is consistent with complete acetylation of the sugar hydroxyl groups. Aliphatic C-H stretching vibrations are observed in the range of 2957-2852cm^{-1} and arise from acetyl methyl groups, the pyranose backbone, and methyl substituents attached to silicon atoms. The absorption band near 1013 cm^{-1} is primarily assigned to the asymmetric Si-O-Si stretching vibration of the trisiloxane fragment. Additional weaker bands observed in the region around 843 cm^{-1} are attributable to Si-C stretching vibrations. The absence of a broad O-H stretching band near 3400 cm^{-1} further confirms the complete acetylation of the molecule.

The chemical composition study of the PMMaPS using ^{13}C NMR **spectrophotometers.** In the ^{13}C NMR spectrum, signals corresponding to methyl (CH_3) groups are observed at δ 20.95 ppm. The acetyl methyl carbons resonate at δ 32.02 ppm. Resonances attributed to the sugar fragment carbons appear in the range of δ 63.02-75.45 ppm. The anomeric carbon of the sugar moiety,

bonded to oxygen, is observed at δ 102.60 ppm. The carbonyl (C=O) carbons of the acetyl groups give rise to signals in the region of δ 169.8-171 ppm.

The chemical composition study of the PMMaPS using ^{1}H NMR **spectrophotometers.** In PMGlPS, seven methyl groups are bonded to silicon, corresponding to a total of 21 protons. In the ^{1}H NMR spectrum, 18 of these protons appear as a singlet at δ 0.16 ppm, while the remaining silicon-bound methyl group resonates as a singlet at δ 0.58 ppm. The methylene protons directly attached to silicon (Si-CH_2) are observed as a multiplet at δ 0.93 ppm.

The acetyl groups give rise to signals corresponding to 12 protons, appearing as a doublet at δ 2.11 ppm. The -O-CH_2- protons (2H) resonate as a triplet at δ 3.48 ppm. Signals from the sugar moiety are observed in the region δ 4.22–5.20 ppm, where one proton appears as a doublet of doublets at δ 4.22 ppm, one proton as a singlet at δ 4.55 ppm, and one proton as a doublet of doublets at δ 5.20 ppm. These resonances are consistent with an acetylated pyranose ring substituted with a trisiloxane fragment.

Scanning electron microscopy of PMMaPS compound. The synthesized PMMaPS was analyzed using scanning electron microscopy (SEM).

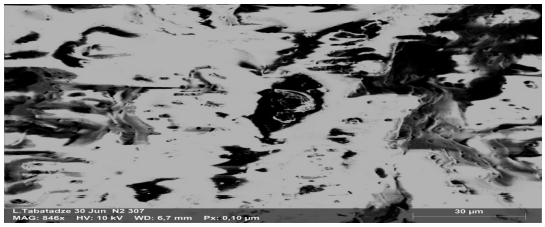


Fig. 1. Scanning electron microscope image of PMMaPS(4)

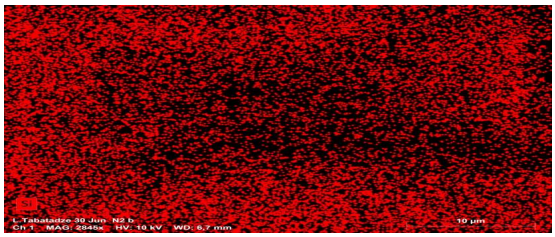


Fig. 2. Element distribution map of PMMaPS (4)

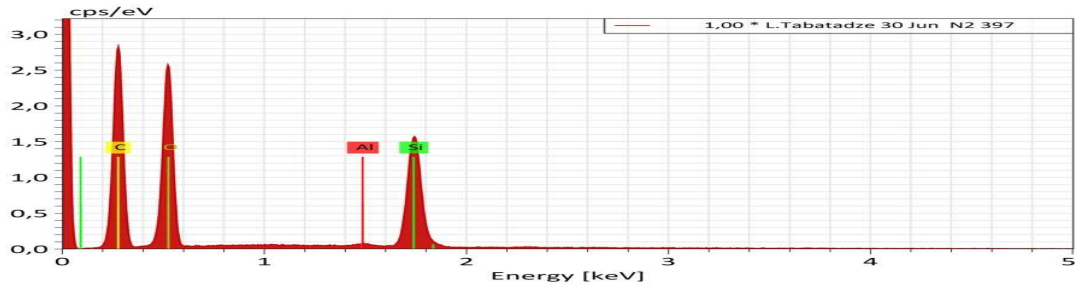


Fig. 3. Substance 4. Elemental analysis of PMMaPS

Element	*At. №*	*Net*	*Mass [%]*	*Mass Norm. [%]*	*Atom [%]*	*Abs. error [mass %]*	*Rel. error [%]*
Carbon	*6*	*66628*	*68,30*	*45,98*	*54,99*	*2,98*	*4,30*

Oxygen	*8*	*62416*	*61,00*	*41,32*	*37,88*	*2,48*	*4,07*
Silicon	*14*	*55481*	*21,45*	*12,49*	*7,03*	*0,85*	*4,17*
Aluminum	*13*	*782*	*0,07*	*0,13*	*0,07*	*0,02*	*10,24*
Sulfur	*16*	*332*	*0,04*	*0,08*	*0,03*	*0,02*	*14,03*
		Sum	*150,86*	*100,00*	*100,00*		

Tab. 1. Substance 4. Elemental analysis of PMMaP

Elemental analysis of PMMaPS was performed using a scanning electron microscope (SEM). The X-ray diffraction results are presented in figure 1-3. and Table 1. Elemental analysis showed that PMMaPS predominantly contained three major elements—carbon (C), oxygen (O), and silicon (Si), while all other detected elements were present only as trace impurities.

Conclusion

Poly[methyl-3-(tetraacetylmannoside)propyl]siloxane for industrial applications was synthesized in high yield by condensation of allyl-2,3,4,6-tetra-O-acetyl-β-D-mannopyranose and polymethylhydrosiloxane (PMHS - a key building block for a wide range of modern silicone materials) using a Pt/C catalyst.

The combined FTIR, ^{1}H NMR, and ^{13}C NMR data unequivocally confirm the successful synthesis and full structural characterization of Overall, of poly [methyl-3- (*tetraacetyl mannoside* side) propyl] siloxane the chemical structure.

The ^{13}C NMR spectrum shows signals for silicon-bound methyl carbons at δ 20.95 ppm and acetyl methyl carbons at δ 20.95 ppm. Resonances corresponding to the sugar ring carbons are observed in the range δ 63.02-75.45 ppm, while the anomeric carbon bonded to oxygen appears at δ 102.60 ppm. The ester carbonyl carbons of the acetyl groups give rise to signals in the region δ 169.8-171 ppm. These chemical shifts confirm complete acetylation of the sugar hydroxyl groups, preservation of the pyranose ring structure, and successful attachment of the heptamethyl trisiloxane moiety.

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