



# Chitin based polyurethanes using hydroxyl terminated polybutadiene. Part I: Molecular engineering



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## ABSTRACT

Chitin based polyurethanes (PUs) using hydroxyl terminated polybutadiene (HTPB) as soft segment were prepared and the structure of the proposed PU was confirmed using FTIR spectrometer. PU prepolymer was prepared using HTPB and toluene-2,4-diisocyanate (TDI), and the chain was extended with different proportions of 1,4-butane diol (BDO) and chitin. During the detailed FTIR study, it was observed that tri-functional character of chitin results in the formation of network structure due to crosslinking of the material, whereas bi-functional aliphatic diol based polyurethane produced linear PU. Hydrogen bonding between the hard segments was identified by the IR spectroscopy. The scanning electron microscope (SEM) analysis also confirmed the cross-linked structure.

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## 1. Introduction

Chitin is the most widespread biopolymer in nature after cellulose. Chitin and its derivatives have great economic value because of their biological activities, and their industrial and biomedical applications. Chitin can be extracted from three sources, namely crustaceans, insects and microorganisms [1]. Chitin is a linear polysaccharide composed of  $\alpha$ -(1-4)-linked 2-acetamido-2-deoxy-D-glucose units which may be de-N-acetylated to some extent. The chitin molecules are known to be ordered into helically micro-fibrillar structures that are embedded into the protein material of the shells. Chitin is closely associated with protein, minerals, and lipids and pigments [2]. Polyurethane elastomers (PUEs) are possibly the most versatile class of polymers due to their biocompatible behavior [3]. Molecular characterization, morphological studies and thermo-mechanical properties of PUEs and PU urea using different diisocyanate, and  $\alpha,\omega$ -alkane diols have been reported by many researchers [3–5]. Polyurethanes functionalized by polysaccharides have been presented in the literature time to time. Synthesis of polyurethanes using sugar [6], chalcone embedded polyurethanes as a biomaterial [7,8] and thermal-responsive chitin-based polyurethane

copolymer as a smart material [9] have been comprehensively reviewed and documented. Surface morphology of starch [10], cellulose [11], and triolein-based PU [12] has also been investigated and well documented. Vegetable oil based PU and PMMA [13], novel bio-antifelting agent based on waterborne polyurethane and cellulose nanocrystals [14], canola oil based PU and PMMA [15] and the role of starch nanocrystals and cellulose whiskers in synergistic reinforcement of waterborne polyurethane [16] have been reported in the literature. Regarding established literature on chitin based polyurethane synthesis, extensive work on structural characterization, crystalline patterns, and thermal properties of chitin-based polyurethane elastomers (PUEs) have been comprehensively reported elsewhere [17–19]. In vitro biocompatibility and cytotoxicity of chitin/1,4-butanediol blend based polyurethane elastomers have been reported in the literature [20–22]. Few reports have been found on the structural characterization of chitin-based polyurethane elastomers and their shape memory characteristics [23–25]. The professional literature and scientific writings have reported possible applications, preparation and properties of hydroxyl terminated polybutadiene based smart materials [26]. Considering excellent biocompatibility and wound healing properties of chitin, intelligent structural properties of hydroxyl terminated polybutadiene and good thermal, elastomeric and biocompatibility of polyurethane, the present project is designed to synthesize chitin based polyurethane using hydroxyl terminated polybutadiene.

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## 2. Experimental

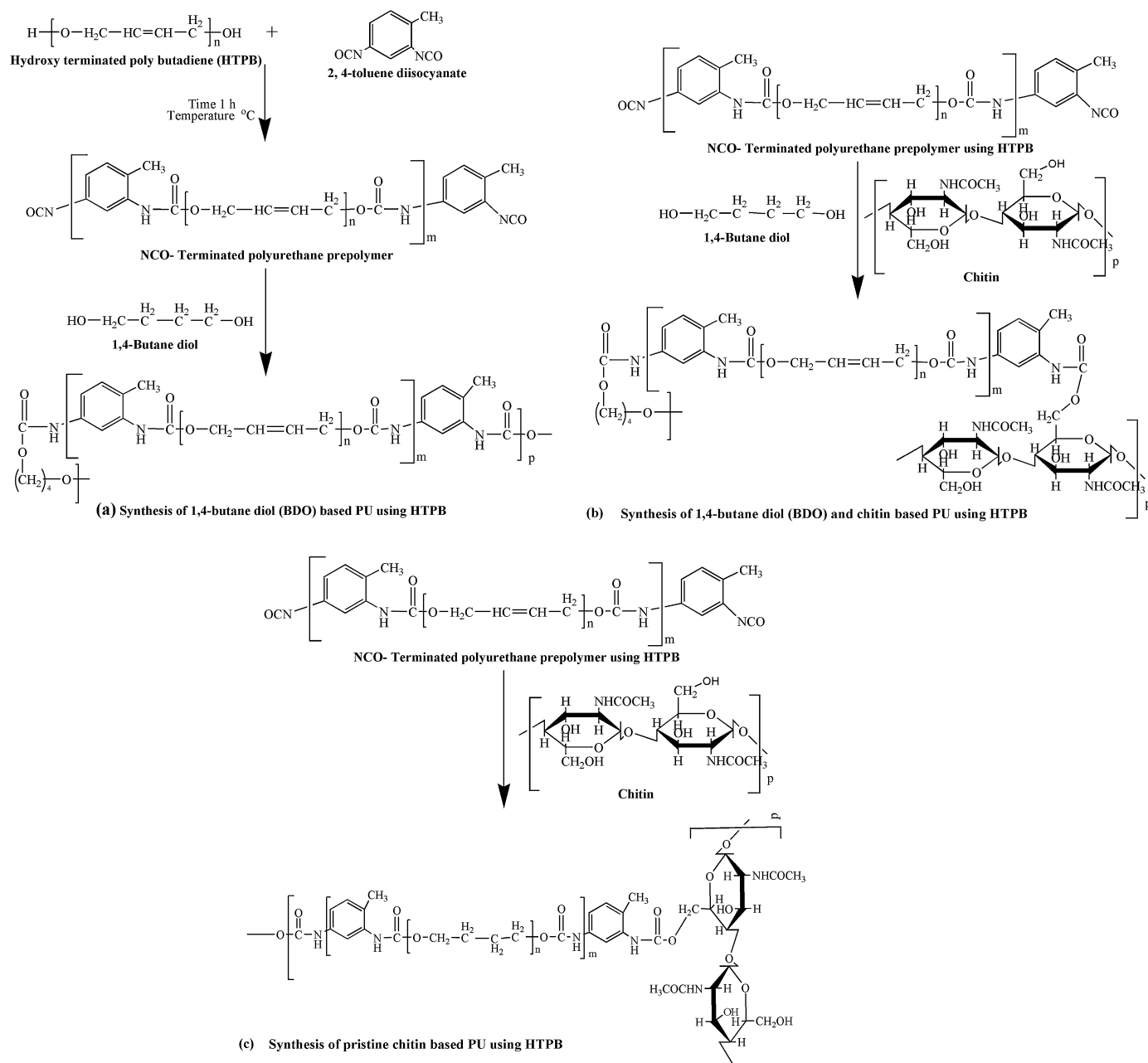
### 2.1. Materials

#### 2.1.1. Chemicals

Toluene diisocyanate (TDI), hydroxyl terminated polybutadiene (HTPB), and 1,4-butane diol (BDO) were purchased from Sigma Chemical Co. (Saint Louis, MO, USA). The flexible spacer i.e., HTPB and the chain extender i.e., BDO used in this study were dried at 80 °C in vacuum for 24 h before use to ensure the removal of all air bubbles and water vapors that may otherwise interfere with the isocyanate reactions. The molecular weight of used soft segment (HTPB) was confirmed by following the procedure reported in ASTM D-4274C [27]. TDI and all of the other materials were used as received. All of the reagents used in this study were of analytical grade.

### 2.2. Synthesis of polyurethane prepolymer using hydroxyl terminated polybutadiene

The synthesis of PU prepolymer was carried out according to recommended procedure [17]. Following the procedures, the PU prepolymer was synthesized by reaction of diisocyanate with diol in ratio of (3:1) to obtain isocyanate (–NCO) terminated polyurethane prepolymer. For this purpose, into a four-necked reaction kettle equipped with mechanical stirrer, heating oil bath, reflux condenser, dropping funnel and N<sub>2</sub> inlet and outlet, polyol (HTPB) was placed. The temperature of the oil bath was increased to 60 °C. Then 2,4-toluene diisocyanate was added and the temperature was then increased to 100 °C. During optimization of the experimental conditions, it was confirmed that the formation of isocyanate (NCO) terminated PU prepolymer completes in 1 h (Fig. 1). A FTIR spectrum of the PU prepolymer was also obtained to confirm the



**Fig. 1.** Synthesis of polyurethane using hydroxyl terminated polybutadiene as a soft segment: (a) 1,4-butane diol (BDO) based polyurethane, (b) 1,4-butane diol (BDO) and chitin based polyurethane, and (c) pristine chitin based polyurethane.

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