Development of high performance polymer nanocomposites using cellulose nanofibrils from isora fibers:- a wood substitute

Thesis submitted to
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in partial fulfillment of the requirements
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Doctor of Philosophy
in
Chemistry

under the Faculty of Science

By
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Under the guidance of
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September 2014

DECLARATION

I hereby declare that the thesis entitled "Development of high performance polymer nano composites using cellulose nanofibrils from isora fibers:- a wood substitute" is an authentic research work carried out by me, under the joint supervision and guidance of Dr. Lovely Mathew, Department of Chemistry, Newman College, Thodupuzha and Prof. (Dr.) Sabu Thomas, School of Chemical Sciences, Mahatma Gandhi University, Kottayam. This work is entirely original in its contents and has not been submitted before either in part or in full to any university or institute for the award of any research degree or diploma.

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Certificate

This is to certify that the thesis entitled **"Development of high performance polymer nano composites using cellulose nanofibrils from isora fibres:-a wood substitute"** is an authentic record of the research work carried out by **Ms. Cintil Jose Chirayil,** under the joint supervision and guidance of Dr. Sabu Thomas (School of Chemical Sciences, M.G University, Kottayam) and myself, in partial fulfillment of the requirements for the award of the degree of **Doctor of Philosophy in Chemistry** under the faculty of Science, Mahatma Gandhi University, Kottayam. The work presented in this thesis has not been submitted for any other degree or diploma earlier. It is also certified that Ms. Cintil Jose Chirayil has fulfilled the course requirements and passed the coursework examination for the Ph.D degree of the university.

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Preface

The development of high performance materials made from natural resources is increasing worldwide and these materials will undoubtedly play a large role in the biocomposite research effort. Among them, cellulose is the most important biopolymer in the new material development. Cellulose fiber reinforced polymer composites have received much attention because of their versatile properties. For the preparation of various types of composites many research works have been performed all over the world on the use of cellulose fibers as filler. This thesis reports the use of isora fiber as a source of cellulose to obtain nanofibrils by steam explosion method. Then these nanofibrils were incorporated into two different polymer matrices, unsaturated polyester (UPR) and plasma modified polyethylene and its properties are studied in detail.

The thesis entitled "Development of high performance polymer nano composites using cellulose nanofibrils from isora fibres:-a wood substitute" consists of nine chapters. First chapter is an updated survey of literature covering the field of cellulose nanofibres, various preparation methods and its modifications. This chapter also describes about different thermoplastic and thermosetting polymers, various aspects of cure reaction, and studies of different nanocomposites. At the end of this chapter the inspiration for this work and the main objectives are explained. The material details, experimental techniques, and characterization methods employed in this study are presented in the second chapter. The third chapter covers the isolation and characterization of cellulose nanofibers from the isora plant using steam explosion technique. Techniques such as FTIR, AFM, high resolution TEM, SEM, XRD, dynamic light scattering and TGA have been used for the characterisation of these nano scale fibers. Finally the properties

of these nanoscale materials have been compared with other nanoscale fibers extracted from various plant sources.

The fourth chapter is focussed on the development of nanocomposite by reinforcing isora nanofibrils (INF) in a thermoset matrix, unsaturated polyester by conventional mechanical mixing process. Of all the thermosetting polymers, unsaturated polyester resins play an important role because of their versatility in properties, flexibility in processing and low cost. In this chapter, scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM) and optical microscopy (OM) have been used for characterising the morphology of nanocomposite. In particular, the nano fibril dispersion and fibril/polyester matrix interactions have been evaluated by studying the mechanical and barrier properties of the nanocomposites. Chapter 5 discusses the rheological and dynamic mechanical behaviour of unsaturated polyester suspensions containing different loadings of isora nanofibril (INF). The interactions were monitored by considering rheological behaviour (liquid state) in comparison with the properties of their matching nanocomposites (solid state). Finally, the amount of polymer chains confined at the nanoculluse net work has been quntified and explaned using qulitiative model.

The 6th chapter deals with the water sorption characteristics of isora nanofibril reinforced polyester composites with special reference to filler loading as well as temperature. The mechanism of diffusion was analysed and the effect of INF on the sorption kinetics was studied. Parameters like diffusion, sorption and permeability coefficients of the composites were determined. Chapter 7 deals with the preparation of nanocomposites using plasma modified polyethylene with different loadings of INFs. The influence of the INF fibrils, on the morphological, mechanical and

viscoelastic properties of polyethylene was studied. Techniques such as SEM, optical microscopy, TEM and AFM have been used for the microscopic studies. The interaction between the filler and matrix has been evaluated by studying the mechanical, dynamic mechanical, rheological and wetting studies of the nanocomposites. Chapter 8 discusses the isothermal crystallization of nanocomposites based on polyethylene as matrix and isora nanofibrils as nanofiller. In order to provide a theoretical basis, the isothermal crystallization kinetics of INF/PE composites has been investigated by differential scanning calorimetry (DSC) at different crystallization temperature (T_c) and the Avrami model was applied to describe the process of isothermal crystallization. The influence of the presence of isora nanofibrils on the kinetic parameters of isothermal crystallization of the matrix polymer and the melting properties of the isothermal crystallized samples has been evaluated. The overall conclusion of the major findings of this research work and future scope are discussed in chapter 9.

CONTENTS

Chapter 2

Materials and Experimental Techniques