In The Name of GOD

Razi University

Razi University
Department of Chemistry

Ph.D. Thesis

Title of the Thesis:

Preparation of modified sulfur composite by using suitable nanofiller and investigate on its properties

Supervisor:

Prof. Mojtaba Shamsipur

Advisor:

Dr. Mohammad sadegh Haji-Tarverdi

By:

Narmin Bahrami Adeh

February 2012

Dedications

This work is dedicated to the memory of my wonderful loving parents Ahmad Bahrami and Hakime Rahimi, my husband Mohsen sadeghi for all the supports he always give me and my lovely children Faeze, Saede and Mohammad Reza.

Without whom none of this would have been possible.

ACKNOWLEDGEMENTS

First of all, I am deeply grateful to Allah (S.W.T.) for his endless blessings and bounties he (S.W.T.) has bestowed upon me.

The list of people that I need to acknowledge in this work is very long. However, time and space constraints limit me to but some people who have been essential to my life and I wish to acknowledge their help at this time.

First and foremost, I am deeply grateful to my supervisor, Dr. Shamsipur, for his support and guidance throughout the course of this dissertation. I have benefited greatly from his knowledge in wide areas of chemistry.

I would like to acknowledge my adviser Dr. Mohammad Sadegh Hajitarverdi. Special thanks are extended to the members of my doctoral committee: Dr. Kazem Kargosha, Dr. Abdolla Yari, Dr. Mohsen Irandoust, Dr. Mohammad Joshaghani, and Dr. Fahimeh Jalali. Your assistance in completing this work is greatly appreciated.

A very special expression of appreciation is extended to Mrs. Fatemeh Zareiee, Mr. Mostafa yazdi, Mr. Nourali Mohammadi, and Hadi Khani for their encouragement, patience, and support.

Finally, my deepest affection and gratitude are owed to my husband Mohsen Sadeghi, and my beloved children Mohammad Reza, Faeze and Saede for their love, support, and sacrifice throughout this work. Without their love and help this endeavor would not have been possible.

Abstract:

In this thesis, sulfur composites were prepared by "Direct mixing" of nanofillers (silica, titania, and clay) with sulfur or plasticized sulfur (sulfur polymer). For plasticizing of sulfur, dicyclopentadiene (DCPD), styrene (St), methyl styrene (MSt) or a mixture of DCPD and St were used. FT-IR spectroscopy was used to characterize the structure of synthesized plasticized sulfur. Mechanical properties, thermal behavior, and morphology study of the prepared composites were also investigated.

The surfaces of nanosilica and nanotitania particles were modified by coupling agents in aqueous and non-aqueous solvent, respectively, and then characterized by FT-IR, TGA, DLS, ZETA-sizer potentiometery, UV–Visible spectroscopy and dispersability tests. Afterwards, the modified nanofillers were incorporated into sulfur/plasticized sulfur to prepare sulfur composites. The thermo-mechanical properties of the samples were tested in order to evaluate the capability of the mentioned "Direct mixing" method. Mechanical properties of the sulfur composite containing microsilica (natural quartz) and nanosilica (fumed) were compared together.

Sulfur composites were synthesized using sodium monmorilloinite (Na-MMT) and organically monmorilloinite (O-MMT) particles by two methods. In the first method, nanoparticles of monmorilloinite was swelling in methyl styrene then were mixed with molten sulfur as swelling procedure. In the second method, without swelling of the particles, the plasticized sulfur was prepared with a "Direct mixing" procedure.

Plasticized sulfur (using methyl styrene) was mixed with various percentages of Na-MMT or O-MMT in order to find the best percentage ratio as critical value by evaluating of the mechanical tests including compressive, flexural, tensile strengths. Also, the nanostructure of the prepared plasticized sulfur-O-MMT composite was confirmed by XRD, TEM, and SEM techniques.

Sulfur with various ratio of DCPD-St mixture was plasticized to find the best mechanical and elongation properties. Then, various percentages of nanosilica (fumed silica) were added to the optimum plasticized sulfur-DCPD-St. The mechanical tests including compressive, flexural and tensile strengths were measured to find the suitable amounts of nanoparticles (critical value).

Also, the effect of mixing rate on the plasticized sulfur-nanosilica composite was investigated by mixing rate of 300 and 10000 rpm and the result showed that changing mixing rate could affect the composite properties.

The prepared plasticized sulfur-nanocomposites were used as coating agents on the surface of concrete in order to evaluate the adhesive ability of the prepared materials.

Overally, the obtained results showed that “Direct mixing” can be used as simple and cheap method for preparing various nanocomposites specially products of commercial importance. The results also showed that modified nanofilres can enhance the mechanical properties of the plasticized sulfur more than unmodified nanofilers.

Keywords: Sulfur, Plasticized sulfur, Modified sulfur, Sulfur composite, Mechanical properties, Nanoclay, Nanosilica, Nanotitania, Nanocomposite