DEVELOPMENT OF RICE HUSK DERIVED NANO MATERIALS HYBRIDIZED WITH POLYANILINE / METAL OXIDES AND COATING ON COTTON FABRIC FOR ENHANCED ULTRAVIOLET SHIELDING

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CERTIFICATE

The research work embodied in the present thesis entitled " **DEVELOPMENT OF RICE HUSK DERIVED NANO MATERIALS HYBRIDIZED WITH POLYANILINE / METAL OXIDES AND COATING ON COTTON FABRIC FOR ENHANCED ULTRAVIOLET SHIELDING**" has been carried out in the Department of Textile Technology, PSG College of Technology, Coimbatore 641004. The work reported herein is original and does not form part of any other thesis or dissertation on the basis of which a degree or award was conferred on an earlier occasion or to any other scholar.

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Abstract

The main objective of the present work is to develop rice husk derivatives such as activated carbon, nanosilica, silicon (Si), silica aerogel (SA) and mesoporous silica (SBA-15) as UV shielding coating materials for cotton fabric. In order to make them as suitable candidates for protective coating against UV radiations, two major protocols were adopted such as grafting and coating. Thus, polyaniline was grafted along with activated carbon or nanosilica over cotton fabric through insitu polymerization approach. In this approach, 3-aminopropytriethoxysilane was used as a linking or crosslinking agent, which hybridize polyaniline and rice husk derivatives (activated carbon or nanosilica) and cotton fabric. Further, titania (TiO_2) was used as dopant in low quantity with silicon, silica aerogel and ceria was doped with SBA-15 to promote their UV shielding behavior. To get the maximum UV shielding, various quantities of these dopants were used and optimization was done. Subsequently, the optimized metal oxide doped rice husk derivatives were suitably surface modified using silane coupling agents, in such a way to favour the compatibility cotton fabric. After, completing the surface functionalization, the rice husk derivatives were coated on cotton fabrics using pad-dry-cure dry method. These developed nanomaterials from rice husk were characterized with different analytical technique such as XRD, SEM, TEM, UV and FTIR spectroscope to ascertain their utility towards UV protective applications. The developed nanomaterials were tested in particle analyser to confirm their existence in nano form. The basic properties like tensile strength, tear strength, bending length and air permeability were studied to assess whether there is any negative impact of treatment given on the cotton fabric. Besides, thermal studies were also performed as an indicator of flame retardancy of cotton fabric.

Initially, the present work deals with the development of amine functionalized activated carbon (FAC) prepared from bio source rice husk and the same has been used

as a UV protective coating material for cotton fabric along with polyaniline network. Thus, different weight percent of both FAC and aniline were used to develop the FAC coated and polyaniline grafted cotton fabric. The resulted cotton fabrics were studied for their UV shielding property. It is quite interesting to note that the UV shielding behavior of developed cotton fabric composite has significantly increased with respect to activated carbon content and hold UPF as 58.7 for 3 wt % FAC coated and 50 wt % aniline. The influence of polyaniline on the shielding behavior was also analyzed by calculating the UV protection factor (UPF) with respect to various concentration of aniline monomer. In the optimization, various percentages of aniline and functionalized activated carbon were tried with the cotton fabric to maximize the UV shielding behavior. As a result of this optimization, it was found that the UPF of the cotton fabric with 3 wt % FAC and 25 wt % aniline increased significantly and reached the maximum 63.9 UPF value. These results show that the activated carbon of rice husk and polyaniline coated cotton fabric composite posses higher UV shielding when compared with that of neat cotton fabric. Thus, the developed material can find better high-performance application as UV shielding material.

In order to utilize another valuable biomass product from rice husk, nanosilica has been derived from it and hybridized with polyaniline through grafting and used as coating material over cotton fabric in subsequent chapter using same insitu approach. In order to optimize the coating percentages of nanosilica and polyaniline, different weight percentage (with respect to the weight of fabric) of both sodium silicate and aniline were used to develop the nanosilica coated and polyaniline grafted cotton fabric. The resulted cotton fabrics were studied for their UV shielding property, thermal and basic fabric properties. Further, scanning electron microscope analysis was performed to ascertain their uniform distribution over the cotton fabric. Interestingly, cotton fabric with 50 % of polyaniline and 0.75 % of silica ($S_{0.75}A_{50}C$) affords the highest UPF as 46.2. These results show that the nanosilica coated and polyaniline grafted cotton fabric composite posses higher UV shielding when compared with that of neat cotton fabric. Thus, this

developed material can also find better high performance application as UV shielding material.

Subsequently, silicon (Si) was prepared via magnesiothermic reduction method and got doped with titania in various concentrations The obtained titania silicon ($Ti_{1-5}Si$) were analyzed for their UV shielding behavior. Further, the optimized Ti_nSi hybrid surface was modified with 3-glycidoxypropyltriethoxysilane to obtain functionalized titania silicon (FTiSi). The developed nanomaterial Ti_nSi was characterized through SEM and TEM. Coating of FTiSi in different weight percentage with respect to cotton was achieved using pad-dry-cure approach. The obtained FTiSi nanoparticles coated cotton fabrics were analyzed for their UV shielding thermal, and basic properties. Further, SEM analysis was performed to reveal the presence and composition of FTiSi nanoparticles over the cotton. The UPF value of 28.2 afforded by the 5 wt % FTiSi coated fabric shows the improved UV shielding tendency. Thus, the titania silicon nanohybrid affords functional behavior to cotton fabric with enhanced UV shielding and thermal stability. Here, it is evident that the utilization of silicon of rice husk with low quantity of titania favors better UV shielding property with retention of inherent fabrics properties.

Further, in pursuit of achievement of our current objective, the UV shielding property of cotton fabric was promoted using silica aerogel doped with minimum amount of titania. Herein, we have prepared the titania silica aerogel ($Ti_{1-15}SA$) from rice husk via sol-gel approach and the optimized sample of Ti_nSA has been surface modified using 3-glycidylpropyltriethoxysilane. Further, the obtained functionalized titania silica aerogel (FTiSA) was coated over cotton fabric using pad-dry-cure method. The obtained cotton fabrics were studied for their UV shielding property, thermal and basic fabric properties in addition to micrograph analysis. Interestingly, it is found that 5 wt % FTiSA coated cotton fabric delivers UPF value of 16.6. These results show that FTiSA coated cotton fabric possesses higher UV shielding when compared to neat cotton fabric. Thus, the developed cotton fabric can find better high performance application as UV shielding material.

Finally, in continuation of our interest, mesoporous silica SBA-15 was prepared from bio-mass waste rice husk and its UV protective behavior was promoted by doping ceria. It is found that, 5 wt. % ceria doped SBA-15 (Ce_5S) shows most extraordinary UV protection behavior. To promote the adhesion of Ce_5S with the surface of cotton fabric, the former has been subjected to surface functionalization using 3-glycidoxypropyltriethoxysilane and denoted as FCeS. Further, different weight percent of FCeS were then coated on cotton fabric using pad-dry cure method. The resulting cotton fabrics were studied for their UV shielding behavior, tensile strength and other basic fabric properties besides thermal stability. The ultraviolet protection factor (UPF) of the cotton fabric coated with 10 wt. % FCeS reached the maximum of 18.9 in addition to the enhanced tensile strength and thermal stability. These results show that the ceria doped SBA-15 coated cotton fabric possess higher UV shielding when compared with that of neat cotton fabric. Thus, the developed low-cost bio-mass material can find better utility as UV shielding material in a wide range of industrial applications.

Thus, in the present work, activated carbon and silica derivatives obtained from biomass rice husk have been used as core and basic coating material to promote the properties of cotton fabric. To achieve the same, appropriate engineering approaches like doping, functionalization and grafting were successfully carried out and are also confirmed with various physico-chemical analyses. Data obtained from various characterization techniques indicates that the enhanced UV shielding and thermal properties of cotton fabrics are due to the coating of nanomaterials of rice husk derivatives. Hence the obtained nanomaterials from rice husks can be used for UV shielding coating, with enhanced strength and thermal property for advanced textile applications.