Abstract

Cellulosic material easily catches fire and generates toxic flammable gases and high temperature during combustion. Different synthetic chemicals are available in the market for making fire retardant cellulosic fabric. However, most of them have been reported as toxic and their requirement in large quantity for delivering flame retardancy make the approach unworthy. Moreover, treatment processes are also cumbersome. Therefore, getting an eco-friendly, cheap, widely available flame retardant material is a big challenge and unmet needs. In this connection, plant extracts have been popularly explored for making functional textiles to impart functionalities like protection against harmful UV rays, bacterial growth and to build aesthetic look by using them as a natural coloring material. In this study, an attempt has been made to explore different wastage plant extract as a novel and green flame retardant for making textile materials fire retardant.

In this research work, three different widely available wastage plant based bio-macromolecules named banana (Musa) peel sap (BPS), coconut (Cocos nucifera) shell extract (CSE) and pomegranate (Punica granatum) rind extract (PRE) have been chosen for the experiment. Primarily, extraction process of the aforementioned bio-macromolecules has been optimized in terms of time and temperature. Thermal stability of the extracted bio-molecules has been examined by different characterization techniques like TGA, EDX, AAS, FTIR, etc. In addition, thesis also elaborates the application of the water based extract and comparison of the potential of fire retardant efficacy of different bio-molecules, once these are applied on cotton textile material. Experimental results on fire retardant efficacy of the treated cotton fabric have been registered systematically in the context. As of summary of the experimental analysis, it has been revealed that the pomegranate rind extract (PRE) has more effectivity towards flame retardancy on cotton fabric as compared to the other two extracts (BPP and CSE). Therefore, on later part of the thesis, PRE has been explored for further in depth study on its flame retardant action on cotton fabric.

PRE has been applied to the cellulosic cotton fabric by padding and exhaust method. Suitable concentration (400g/L) of application of PRE has been optimized on the basis of the Limiting Oxygen Index (LOI) value of the treated fabric (32). For application of PRE on cotton fabric, different treatment processes [padding (both cold and hot), exhaust with varying time, temperature and material to liquor ratio] have been explored. In each and every application process, add-on% of the treated fabric has been recorded. Moreover, fire retardancy of all the treated fabrics has been examined in terms of LOI value and the vertical flammability test. It has been noticed that the hot padding method is the best approach for the uniform application of PRE on the cotton fabric. Apart from the hot padding method, it has also been mentioned that the exhaust process is also suitable for the uniform application, if it is being carried out in a dyeing machine where both the fabric and the liquor are in circulation. Thermal stability of the treated fabric has also been evaluated by forced combustion test. Both the control and treated cotton fabric have been combusted in cone calorimeter machine with a heat flux level of 35kW/m2 which is suitable for the upholstery, curtain like (home-furnishing) materials. It has been observed that the average peak heat release rate of the treated fabric (treated by 400g/L PRE) is almost half (45kW/m2) compared to the peak heat release rate of the control cotton fabric (80kW/m2).

Thermo-gravimetry test of the same treated fabric illustrates earlier dehydration and more amount of char mass formation at the end of the test. The resulting fire retardancy of the treated fabric is the combined reaction effect of the acid source (carbamic acid, ammonium salt, hexacontanoic acid, etc.), phenolic source (poly phenolic tannin, gallic acid, ellagic acid, betacyanin, coumarin, etc.) carbon source (carbonic dihydrazide, nona hexacontanoic acid, 1 hydroxy 2 pentanone, sugar based material), blowing agent, Nitrogen containing bases like guanidine, asparagine (amino acid of protein), etc. present in the PRE extracts as observed from the Gas Chromatography Mass Spectroscopy (GC-MS) and Liquid Crystal Mass Spectroscopy (LC-MS) analysis. FTIR analysis of the char mass (char of PRE
treated fabric) shows structural stability and aromatized cellulosic structure. Surface morphological analysis of the char mass of the treated fabric displays structural integrity (weaves are present clearly like control cotton) with a presence of small bubbles on its surface. On the contrary, char mass of the control cotton fabric shows grey colour, net like fragile structure. Energy dispersive X-ray (EDX) analysis of the treated fabric char reveals the presence of Carbon element which is another positive indication of the aromatized structure. For further confirmation, connected with the flammable gas formation, volatile species coming out during burning of the treated fabric also have been analyzed by GC-MS. It has been observed that the peaks (mass/charge) assigned with the levoglucosan and its derivatives (77, 65, 91, 162) are very less intensive in the smoke of the treated fabric whereas smoke of the control cotton fabric shows levoglucosan based components and tar like products, terpenoids, etc. For understanding the char formation mechanism, solid NMR analysis of the char mass of the control and treated fabric also has been performed and discussed in detail in the concern chapter. It has been noticed that PRE helps in aromatization of the cellulosic based cotton fabric in increasing the Carbon content in char mass and restricts the flammable gas formation at higher temperature.

PRE treated fabric shows afterglow duration of more than 200s and the add-on% required for the self-extinguishment is also more than 35%. Therefore, for reducing the extent of afterglow and the add-on%, a mixed formulation of the pomegranate rind extract (PRE) and Sodium tri polyphosphate (STPP) has been integrated into cotton fabric to evaluate its efficacy of flame retardancy. It has been found that the control, 3% (w/v) STPP and PRE treated cotton fabric register LOI value of 18, 21 and 26, respectively and completely burns within 80, 120 and 420s, respectively in the vertical flammability test. On the contrary, (PRE+ 3% STPP) treated cotton fabric exhibits the LOI value of 35 with a specific char length of 30mm. Extent of afterglow duration on the treated fabric has been found to be around 70-80s. Thermo-gravimetry (TG) analysis of the control, PRE and 3% STPP treated cotton fabric shows only 1-2 % remaining char mass at 550°C whereas the (PRE+ 3% STPP) treated fabric registers the char mass retention of more than 10% at the said temperature. Cone calorimetric data reveals that the peak heat release rate (PHRR) for the treated fabric is 25kW/m² (almost one third) compared to the heat release value of the control cotton fabric (79kW/m²).

At last part of the thesis, some experimental works have been investigated to improve the wash durability of the treatment on cotton fabric. Corroborated with the wash durability, different carboxylic acid based compounds Butane Tetra Carboxylic Acid (BTCA), Citric Acid (CA) have been explored. It has been found that 10% citric acid formulation based PRE treatment on the cotton fabric assists to achieve the LOI value of 26 after one single ISO-2 washing in launder-o-meter machine, while BTCA treatment registers the similar LOI value of 26-27 after single washing by following same washing method.