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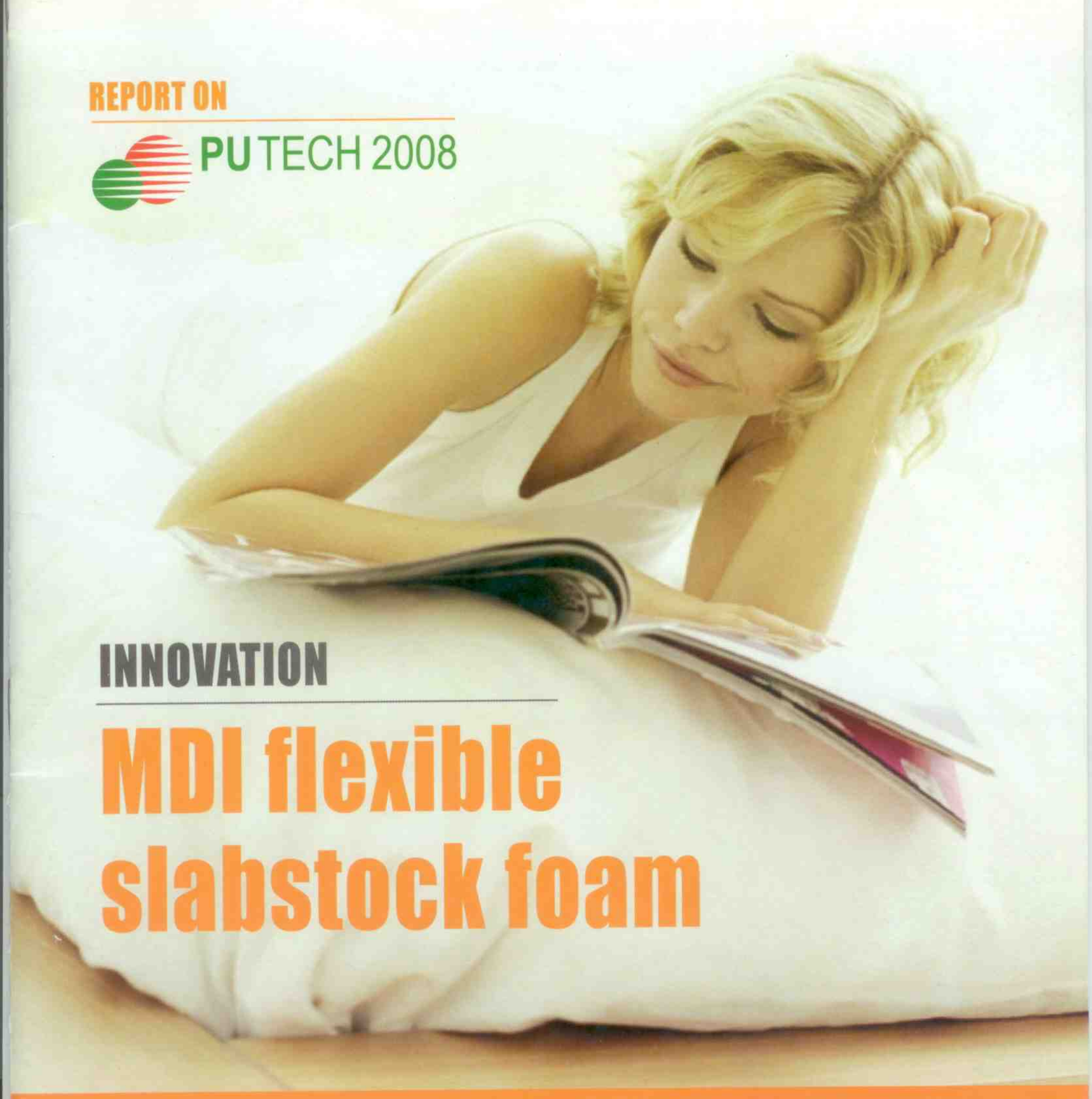

REPORT ON

PUTECH 2008

INNOVATION

MDI flexible slabstock foam

Dr. Harpal Singh

A Profile of a Scientist: 'Fire Retardant Rigid Polyurethane Foam'

Dr. Harpal Singh is holding a M.Sc. degree in Organic Chemistry, and has recently received his Ph.D. in Fire Retardant Rigid Polyurethane Foam, from the Indian Institute of Technology Roorkee. He began his career as a research scientist in 1990, at the Central Building Research Institute, Roorkee in the department of Fire Research. He has considerable experience in fire retardancy of combustible building materials. Since 1996, he is working on cellular plastics and polymers and particularly on rigid polyurethane, foam to render it fire retardant. He has authored more than 25 research papers in International as well as National Journals and Conferences.

A Summary of his Ph.D. Thesis: "Studies on Flame Inhibition for Imparting Flame Retardancy in Polymeric Rigid Foam."

Under the category of polymeric rigid foam, rigid polyurethane foam (RPUF) was selected as the research problem. RPUF is one of the most versatile and widely used thermal insulation materials throughout the world. Great utilities of RPUF as thermal insulation material are due to its properties such as low thermal conductivity, low density, high strength to weight ratio and low moisture permeability. Besides thermal insulation, it also finds many applications in transportation, automotive industry, packaging and refrigeration technology and appliances etc. RPUF, being a highly cellular polymer is easily ignitable and highly flammable. On burning it undergoes decomposition and produces large quantity of vision obscuring smoke with highly toxic gases such as hydrogen cyanide, carbon monoxide etc. Thus it is important to render it flame retardant for safe use. A lot of research work for rendering RPUF fire retardant is reported in the literature. RPUF is rendered fire retardant generally through additive and reactive approaches. Very little work has been reported in the literature to render RPUF fire retardant through impregnation and coating. Reactive and additive approaches are considered better than impregnation and coating. However, fire retardant additives and approaches used so far have some limitations in terms of flammability characteristics (extent burnt, burning rate, percent mass loss, extinction time and oxygen index) and smoke density. Thus, there is ample scope and an urgent need to develop rigid polyurethane foam with a reduced level of flammability and smoke.
The main objective of the present research is to first prepare a chemical formulation for RPUF. The reactivity of various foam ingredients and their effects on the physical properties of foam such as cream time, gel time, tack-free time and density are studied. Subsequently, prepared RPUF is rendered fire retardant through additive and combination of additives incorporated at the formulation stage and, impregnation and coating at a later stage. The resultant RPUF exhibits a better level of flame retardancy with reduced smoke density.

Formulation of RPUF based on crude 4, 4'-diphenylmethane diisocyanate (CMDI), polyether polyol, triethylene diamine (TED), 1, 4-butane diol (BD), polysiloxane ether, methylene chloride and water has been studied. The stoichiometric ratio of various foam ingredients and their effects on the physical properties such as cream time, gel time, tack-free time and density of the RPUF samples has been studied. The results indicate that the rate of RPUF formation was increased with the catalyst (TED, tin) and water contents. The density of RPUF samples blown by water, methylene chloride and mixture of water and methylene chloride was decreased from 240.1 to 33.4 kg/m^3 with an increase of blowing agent contents. However, the density of RPUF was increased with increasing content of 1, 4-butane diol. The cell morphology and thermal properties of the RPUF samples were investigated with scanning electron microscopy (SEM), thermogravimetric analysis (TGA), derivative thermogravimetry (DTG) and differential thermal analysis (DTA). SEM results exhibit the average increase in the cell size of the RPUF samples from 162 to 278 mm with the increased water content. Thermal behaviour study indicates that RPUF samples were decomposed in nitrogen and degraded in air through two and three weight loss stages respectively. Foam pyrolysis in nitrogen and combustion in air lead to 15% and 0% char residue respectively. The results indicate that thermal stability of RPUF is relatively better in nitrogen than in air atmosphere.

Three approaches were used to render prepared RPUF fire retardant i.e. additives incorporation at the formulation stage and, impregnation and coating with compositions at a later stage. In the first approach, RPUF was incorporated with ATH, melamine (M), combination of ATH and melamine additives at the formulation stage. In the second and third approaches, RPUF was impregnated with PUMF composition and, coated with phosphoric acid (P), chlorub (Cl) and combination of phosphoric acid and chlorub (P-Cl) composition. The density of RPUF with different additives was studied with various additive contents. The density of additives incorporated, impregnated and coated RPUF was determined as per ASTM D1622. The density of RPUF-ATH and RPUF-M is increased from 49.19 to 106.18 kg/m^3 with an increase of ATH and melamine contents. The density of impregnated and coated RPUF increased to 80.17 and 57.68 kg/m^3 respectively with an increase of respective composition concentrations.

In order to understand the effect of additives on morphology, elemental presence and thermal properties of RPUF, the properties were investigated with scanning electron microscopy (SEM), CHNS analyzer and energy dispersive X-rays (EDX) and, TGA/DTG/DTA. The micrographs of RPUF-ATH, RPUF-M and RPUF-ATH-M show the ATH and melamine as white particles are dispersed everywhere in the foam skeleton. PMUF impregnation decreases the cell size from 162 to 123 ìm due to the PMUF deposition on cell walls and surfaces. The SEM images of RPUF-P, RPUF-Cl and RPUF-P-Cl exhibit the presence of thin white layer spread on the cell walls and surfaces. Elemental analysis of RPUF shows the presence of carbon, hydrogen, oxygen and nitrogen. The elemental presence of aluminum and enhanced percentage of nitrogen, phosphorus and enhanced nitrogen percentage and, phosphorus and chlorine were added to the RPUF incorporated with ATH and melamine, impregnated with PMUF and coated with phosphoric acid and chlorub respectively. Thermal behaviour study indicates that RPUF and additives added RPUF samples decomposed in nitrogen and degraded in air through two and three weight loss stages respectively. Thus degradation is more complex in air than nitrogen. The on-set degradation temperatures and the quantity of char residue were relatively high in nitrogen than air. It shows that the thermal stability of both foams was relatively better in nitrogen than in air atmosphere. However, relatively lower on-set degradation temperature and high quantity of char residue produced by additives added RPUF show that they can perform better at elevated temperature than conventional RPUF.

Flammability performance, oxygen index and smoke density of RPUF and additives added RPUF samples were investigated with BS: 4735, ASTM D2863 and ASTM E662 respectively. RPUF shows maximum extent burnt, burning rate and percent mass loss (PML) under flammability test. RPUF incorporated with ATH and melamine, impregnated with PMUF and coated with phosphoric acid and chlorub exhibit great decrease in extent burnt, burning rate and PML. The flammability of additives added RPUF samples decreased with an increase of additive contents. RPUF samples incorporated with ATH and melamine exhibit better fire performance than PMUF impregnated and phosphoric acid-chlorub coated samples. The oxygen index of RPUF samples increased from 17.8 to 27.2, 24.1 and 32.9 when the maximum contents of ATH and melamine, PMUF and phosphoric acid-chlorub are added respectively. The oxygen index value indicates that ATH and melamine incorporated RPUF samples exhibit better fire retardancy than PMUF impregnated and phosphoric acid - chlorub coated samples. The order of smoke density was slightly different from the order of flammability performance and oxygen index of additives added RPUF. RPUF generates early and maximum smoke, whereas, additives added RPUF generates delayed and less smoke under flaming and non-flaming mode. However, smoke density was more under flaming than non-flaming mode. RPUF-ATH generates minimum smoke, whereas, RPUF-P generates slightly maximum smoke under flaming and non-flaming mode. Although RPUF-P generates more smoke than RPUF under flaming and non-flaming mode, however, the trend of smoke generation was entirely different. The maximum smoke was generated in the end of the exposure.

Thus, it has been possible to render RPUF flame retardant to substantial extent by additives incorporation, impregnation and coating. The best flame retardancy is exhibited by additives incorporated and followed by impregnated and coated RPUF. A comparison of developed RPUF with the reported RPUF in terms of fire retardancy shows that developed RPUF exhibits improved fire retardancy with reduced level of smoke density. Thus, the present research work on RPUF has resulted in the development of fire retardant RPUF with reduced level of smoke density which can be used safely in many applications.