

CONTENTS

|  | <b>Page No.</b> |
|--|-----------------|
| <i>Title Page</i>  | <i>i</i>        |
| <i>Dedication</i>  | <i>ii</i>       |
| <i>Certificate of Approval</i>                             | <i>iii</i>      |
| <i>Certificate by the Supervisors</i>                      | <i>iv</i>       |
| <i>Acknowledgements</i>                                    | <i>v</i>        |
| <i>Declaration</i>   | <i>vi</i>       |
| <i>List of Symbols</i>                                     | <i>vii</i>      |
| <i>List of abbreviations</i>                               | <i>viii</i>     |
| <i>Abstract</i>  | <i>xi</i>       |
| <i>Contents</i>  | <i>xii</i>      |
| <br>   |                 |
| <b>CHAPTER 1 Introduction</b>                              | <b>1–31</b>     |
| 1.1 Polymeric foams  | 1               |
| 1.1.1 Historical development of polymeric foams            | 2               |
| 1.1.2 Basic principles in the formation of polymeric foams | 3               |
| 1.1.3 Application of polymeric foams                       | 4               |
| 1.2 Polyurethanes (PUs)                                    | 4               |
| 1.2.1 Polyurethane foams (PUFs)                            | 8               |
| 1.2.1.1 Flexible polyurethane foam                         | 9               |
| 1.2.1.2 Rigid polyurethane foam                            | 10              |
| 1.2.1.2.1 Polyols  | 11              |
| 1.2.1.2.2 Isocyanates                                      | 13              |
| 1.2.1.2.3 Blowing agents                                   | 13              |
| 1.2.1.2.4 Catalysts  | 15              |
| 1.2.1.2.5 Surfactants                                      | 16              |
| 1.3 Flame retardants (FRs)                                 | 17              |
| 1.3.1 Selection and requirements for FRs                   | 19              |
| 1.3.2 Thermal decomposition mechanism                      | 20              |
| 1.3.3 Polymer combustion process                           | 21              |
| 1.3.4 Inhibition of polymer combustion                     | 22              |
| 1.3.4.1 Halogenated FR additives                           | 23              |
| 1.3.4.2 FR additives based on phosphorus                   | 24              |
| 1.3.4.3 Inorganic hydroxide FR additive                    | 25              |
| 1.3.4.4 FR additives based on nitrogen                     | 25              |
| 1.3.4.5 Intumescent char FR additives                      | 26              |

|                  |   |              |
|------------------|---|--------------|
| 1.3.4.6          | FR additives based on nanoclay                  | 27           |
| 1.4              | Flame retardant rigid polyurethane foam         | 28           |
| 1.5              | Applications of rigid polyurethane foam         | 29           |
| 1.6              | Scope and objective of the present work         | 30           |
| <b>CHAPTER 2</b> | <b>Experimental Section</b>                     | <b>33–44</b> |
| 2.1              | Introduction                                    | 33           |
| 2.2              | Materials                                       | 33           |
| 2.2.1            | Raw materials                                   | 33           |
| 2.2.1.1          | Polyols   | 33           |
| 2.2.1.2          | Isocyanate                                      | 33           |
| 2.2.1.3          | Catalysts                                       | 33           |
| 2.2.1.4          | Blowing agents                                  | 33           |
| 2.2.1.5          | Surfactant                                      | 34           |
| 2.2.1.6          | Other chemicals                                 | 34           |
| 2.2.2            | Fillers   | 34           |
| 2.2.3            | Flame retardant (FR) additives                  | 34           |
| 2.2.3.1          | Phosphorus based FR additives                   | 34           |
| 2.2.3.2          | Inorganic hydroxide based FR additive           | 35           |
| 2.2.3.3          | Nitrogen based FR additives                     | 35           |
| 2.2.3.4          | Intumescent char based FR additive              | 35           |
| 2.2.3.5          | Other additives                                 | 35           |
| 2.3              | Preparation of rigid polyurethane foam          | 36           |
| 2.3.1            | Calculations                                    | 37           |
| 2.4              | Testing of rigid polyurethane foam              | 37           |
| 2.4.1            | Viscosity                                       | 37           |
| 2.4.2            | Density   | 38           |
| 2.4.3            | Compressive strength and modulus                | 38           |
| 2.4.4            | Thermal conductivity                            | 39           |
| 2.4.5            | Fourier transform infrared spectroscopy (FT–IR) | 39           |
| 2.4.6            | Scanning electron microscopy (SEM)              | 39           |
| 2.4.7            | Wide angle X–ray diffraction (WAXD)             | 40           |
| 2.4.8            | Transmission electron microscopy (TEM)          | 40           |
| 2.4.9            | Differential scanning calorimetry (DSC)         | 40           |

|                  |   |               |
|------------------|---|---------------|
|                  | 2.4.10 Thermogravimetric analysis (TGA)                 | 40            |
| 2.5              | Testing of fire retardant properties of PUF             | 41            |
|                  | 2.5.1 Limiting oxygen index (LOI) test                  | 41            |
|                  | 2.5.2 Flame spread rate                                 | 41            |
|                  | 2.5.3 Cone calorimeter test                             | 42            |
|                  | 2.5.4 Smoke density measurements                        | 42            |
|                  | 2.5.5 Toxicity analysis                                 | 43            |
|                  | 2.5.6 Char yield measurement                            | 44            |
| <b>CHAPTER 3</b> | <b>Effect of Foam Density on the Properties of PUF</b>  | <b>45–53</b>  |
| 3.1              | Introduction  | 45            |
| 3.2              | Results and Discussion                                  | 45            |
|                  | 3.2.1 Mechanical properties                             | 47            |
|                  | 3.2.2 Morphology  | 50            |
|                  | 3.2.3 Thermal conductivity                              | 51            |
|                  | 3.2.4 Thermal analysis                                  | 52            |
| 3.3              | Conclusions   | 52            |
| <b>CHAPTER 4</b> | <b>Effect of Raw Materials on the Properties of PUF</b> | <b>55–75</b>  |
| 4.1              | Introduction  | 55            |
| 4.2              | Effect of types of polyols and their blends             | 55            |
| 4.3              | Effect of concentration of chain extender               | 57            |
| 4.4              | Effect of types and concentration of catalysts          | 61            |
| 4.5              | Effect of mixed blowing agents                          | 65            |
| 4.6              | Effect of concentration of surfactant                   | 68            |
| 4.7              | Effect of isocyanate index                              | 71            |
| 4.8              | Conclusions   | 74            |
| <b>CHAPTER 5</b> | <b>Effect of Fillers on the Properties of PUF</b>       | <b>77–102</b> |
| 5.1              | Introduction  | 77            |
| 5.2              | Results and Discussion                                  | 79            |
|                  | 5.2.1 Effect of calcium carbonate and glass powder      | 79            |
|                  | 5.2.2 Effect of silica                                  | 84            |
|                  | 5.2.3 Effect of nanoclays                               | 90            |
| 5.3              | Conclusions   | 102           |

|                  |   |                |
|------------------|---|----------------|
| <b>CHAPTER 6</b> | <b>Thermal and Fire retardant Properties of PUF using Conventional FR Additives</b> | <b>103–140</b> |
| 6.1              | Thermal degradation of PUF under inert and air atmosphere                           | 103            |
| 6.2              | Effect of phosphorus based FR additives   | 107            |
| 6.2.1            | Thermal analysis  | 109            |
| 6.2.2            | Flame retardant properties  | 113            |
| 6.3              | Effect of alumina trihydrate (ATH)  | 119            |
| 6.3.1            | Physico-mechanical properties   | 121            |
| 6.3.2            | Thermal analysis  | 124            |
| 6.3.3            | Flame retardant properties  | 127            |
| 6.4              | Effect of nitrogen based FR additives   | 130            |
| 6.4.1            | Thermal analysis  | 132            |
| 6.4.2            | Flame retardant properties  | 134            |
| 6.4.3            | Flame retardant mechanism   | 138            |
| 6.5              | Conclusions   | 140            |
| <b>CHAPTER 7</b> | <b>Intumescent Flame Retardant PUF Prepared by using Expandable Graphite (EG)</b>   | <b>141–155</b> |
| 7.1              | Introduction  | 141            |
| 7.2              | Results and Discussion  | 143            |
| 7.2.1            | Physico-mechanical properties   | 143            |
| 7.2.2            | Thermal analysis  | 148            |
| 7.2.3            | Flame retardant properties  | 150            |
| 7.3              | Conclusions   | 155            |
| <b>CHAPTER 8</b> | <b>Fire Retardant High Density PUF Prepared by Molding Method</b>                   | <b>157–163</b> |
| 8.1              | Introduction  | 157            |
| 8.2              | Results and Discussion  | 158            |
| 8.3              | Conclusions   | 163            |
| <b>CHAPTER 9</b> | <b>Summary and Conclusions</b>  | <b>165–167</b> |
| 9.1              | Summary and conclusions   | 165            |
| 9.2              | Contribution of the thesis  | 167            |
| 9.3              | Future scope of study   | 167            |

|                             |                |
|-----------------------------|----------------|
| <b>REFERENCES</b>           | <b>169–200</b> |
| <b>CURRICULUM VITAE</b>     |                |
| <b>LIST OF PUBLICATIONS</b> |                |

Copyright  
IIT Kharagpur