

CHAPTER V

CONCLUSION

Five polyphosphonates and ten polyphosphates were successfully synthesized by low-temperature solution polycondensation from various combinations of five bisphenols with phenylphosphonic dichloride and two *p*-substituted phenylphosphorodichloridates. The optimized condition was obtained when dichloromethane was used as solvent and with high monomer concentration (0.01 mol bisphenol in 20 mL dichloromethane and 0.01 mol of phenylphosphonic dichloride or *p*-substituted phenyl-phosphorodichloridate in 5 mL of dichloromethane) in the presence of triethylamine at reflux for 1h. The weight average molecular weights of polymers lie between 8,500 and 95,000 Dalton. The structure of polymers are confirmed spectroscopically (¹H-NMR, ¹³C-NMR, ³¹P-NMR, IR). All polymers degrade between 436 and 494 °C. The glass transition temperature values of polyphosphonates are higher than those of polyphosphates. The incorporation of bromine atoms into the polymer chain leads to high glass transition temperature, low degradation temperature and increased flame retardancy. LOI value of the polymers are between 28 and 40. The correlation of percent P with LOI and percent char yield (at 700 °C) are in the similar trend. The polymers with higher percent P led to higher percent char yield and LOI values. All results of polymer characterizations are summarized in **Table 5.1**.

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Table 5.1 Summarized results of all synthesized polymers

| Polymer | % yield | \bar{M}_w (D) | T_g (°C) | T_d (°C) | % char at 700 °C | LOI |
|----------------------------|---------|-----------------|---------------|---------------|---------------------|----------------|
| polyphosphonate 16a | 52 | 14,346 | 99.0 | 476.0 | 22 | 28.0 |
| polyphosphonate 16b | 52 | 8,481 | 120.2 | 466.8 | 13 | - ^a |
| polyphosphonate 16c | 63 | 15,275 | 94.4 | 484.5 | 35 | 34.3 |
| polyphosphonate 16d | 59 | 31,269 | 84.9 | 482.2 | 18 | 26.4 |
| polyphosphonate 16e | 52 | 11,906 | 113.7 | 465.2 | 15 | 26.2 |
| polyphosphate 19a | 46 | 9,332 | 72.3 | 448.8 | 18 | 32.9 |
| polyphosphate 19b | 70 | 14,892 | 85.4 | 445.4 | 12 | 34.5 |
| polyphosphate 19c | 46 | 16,390 | 66.2 | 462.9 | 26 | 40.8 |
| polyphosphate 19d | 70 | 63,357 | 64.9 | 466.4 | 13 | 36.4 |
| polyphosphate 19e | 53 | 9,295 | 85.6 | 447.7 | 16 | 37.1 |
| polyphosphate 20a | 52 | 19,386 | 61.1 | 470.0 | 22 | 27.2 |
| polyphosphate 20b | 63 | 12,895 | 76.3 | 449.9 | 13 | 30.4 |
| polyphosphate 20c | 54 | 94,817 | 55.4 | 494.4 | 24 | 34.6 |
| polyphosphate 20d | 69 | 24,408 | 54.9 | 469.4 | 17 | 33.2 |
| polyphosphate 20e | 56 | 14,204 | 80.4 | 436.7 | 13 | 32.8 |

^aNot characterized.

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