酸源与气源配比对膨胀阻燃剂炭层形成和阻燃性能的影响

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摘要：【目的】为探究酸源与气源比例与膨胀型阻燃剂阻燃效果之间的关系，并分析其对阻燃处理杨木燃烧特性的影响。【方法】选用植酸和尿素分别作为酸源和气源，制备比例为 1:3、1:6、1:10、1:12 的膨胀型阻燃剂，即 PAN-3、PAN-6、PAN-10、PAN-12 膨胀型阻燃剂，并采用全自动元素分析仪验证分析上述 4 种阻燃剂中 N 元素与 P 元素的比例。采用真空浸渍法获得阻燃处理杨木试样，借助热重（TGA）、锥形量热（CONE）分析其化学组分变化以及热降解和燃烧特性，采用扫描电镜观察 CONE 测试后木材残炭的微观形貌，结合激光拉曼显微镜与 X 射线衍射仪（XRD）对残炭的石墨化程度进行分析。【结果】随着气源比例的增加（气源:酸源≥3），阻燃处理杨木 TG 曲线中的最大降解温度向高温方向移动，燃烧时会形成热稳定性较好的膨胀炭层结构，木材残炭的细胞壁厚度呈现先上升后下降的趋势，且残炭的石墨化程度在不断增加，这意味着膨胀结构在燃烧期间对热释放和质量损失的抑制作用更大。当气源比例的增加到一定程度（气源：酸源≥10），过量气源导致膨胀炭层结构破裂，其隔热、隔氧性能下降。CONE 结果显示，经过阻燃处理试样的总热释放量与热释放速率峰值相较于未处理组均有所下降，其中 PAN-6 组的降幅达到 63.97%与 64.1%。气源在 103□-193□分解产生氨气，酸源在 193□-291□分解产生偏磷酸催化木材脱水成炭，随着气源比例增加，两者协同作用下生成的炭层高稳定性和完整性增加，降低了阻燃杨木的热释放，同时增强了炭层对烟气和一氧化碳释放的抑制作用，其中 PAN-6 组烟气释放量下降了 74.31%、热释放量下降了 39.79%。当膨胀炭层完整性遭到破坏时，PAN-12 组烟气释放量增加了 17.58%、热释放量增加了 2.53%。【结论】初步得出随着气源添加，会生成高完整性、高稳定性的膨胀炭层，膨胀型阻燃剂的阻燃、抑烟性能得到改善；但当气源：酸源比值大于 10 时，阻燃炭层的完整性遭到破坏促使阻燃处理木材的热释放量与烟气释放量均增加。

关键词：膨胀型阻燃剂；杨木；阻燃；炭层形成

Effect of acid and gas source ratio on char layer formation and flame retardancy of the intumescent flame retardant

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Abstract:【Objective】This study aims to investigate the correlation between the acid and gas source ratio and the

flame retardant effect of intumescent flame retardants (IFR), and analyze their impact on the combustion characteristics of flame-retardant-treated poplar wood (FW).【Method】Phytic acid and urea were selected as acid source and gas source, respectively. IFRs with different ratios (1:3, 1:6, 1:10, and 1:12) were prepared and labeled as PAN-3, PAN-6, PAN-10, and PAN-12. The ratio of nitrogen and phosphorus elements in the four IFRs were verified and analyzed with a fully automatic elemental analyzer. FW samples were obtained through vacuum impregnation, and their thermal degradation and combustion characteristics were analyzed by thermogravimetric analysis (TGA) and cone calorimetry (CONE). Scanning electron microscopy (SEM), laser Raman microscopy, and X-ray diffraction (XRD) were used to investigate the microscopic morphology and degree of graphitization of wood char residues.【Result】 As gas source content increased (gas source: acid source≥3), the expanded structure of the flame-retardant poplar wood promoted the higher temperature shift of the maximum degradation temperature on the TGA curve and formed a more thermally stable char layer. Additionally, the thickness of the cell wall of the char residue showed an initial increase and then a decrease. It was found that the inhibitory effect of the expanded char structure on heat release and mass loss was greater with the addition of gas source, which was reflected in the continuous increase of the degree of graphitization of the char residue. When the gas source ratio increased to certain degree (gas source: acid source ≥10), an excess of gas source resulted in the rupture of the expanded structure, and a decrease in the thermal insulation and oxygen insulation properties.

CONE results showed that the total heat release (THR) and the peak of heat release rate (PHRR) of the FW samples decreased compared with that of the untreated group, wherein the reduction of the PAN-6 group reached 63.97% and 64.1%. Ammonia was produced by the gas source decomposition at 103℃-193℃, and the acid source was transformed into pyrophosphate, catalyzing wood dehydration into char at 193℃-291℃. A high stability and integrity char layer was formed based on the synergistic effect of gas and acid source, which had a inhibitory effect on the heat release, as well ad smoke and carbon monoxide release. The PAN-6 group showed a decrease of 63.97% and 64.1% for THR and PHRR, respectively. However, when the integrity of the expanded char layer was destroyed, the PAN-12 group showed an increase of 17.58% and 2.53% for smoke release and heat release, respectively.【Conclusion】The addition of gas source could produce a char layer with high integrity and stability, improving the flame retardancy and smoke suppression properties of the IFRs. However, when the gas and acid source ratio is higher than 10, the destruction of the char layer's integrity increased in heat release and smoke release of the FW.

Keywords: intumescent flame retardant, poplar wood, flame retardancy, char layer formation