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热重分析-单滴微萃取-气相色谱-质谱结合傅里叶变换红外光谱考察咖啡酸的热解行为

杨继1,2, 杨柳1*, 朱文辉1,3, 吴亿勤1, 曹秋娥2

1. 红塔烟草(集团)有限责任公司技术中心, 云南 玉溪 653100; 2. 云南大学化学科学与工程学院, 云南 昆明 650091; 3. 云南民族大学化学与生物技术学院, 云南 昆明 650031

Investigation of pyrolysis process of caffeic acid in nitrogen using thermogravimetry-single drop microextraction-gas chromatography-mass spectrometry and Fourier transform infrared spectroscopy

YANG Ji1,2, YANG Liu1*, ZHU Wenhui1,3, WU Yiqin1, CAO Qiue2

1. Research and Development Center of Hongta Tobacco (Group) Co., Ltd., Yuxi 653100, China; 2. College of Chemistry and Chemical Engineering, Yunnan University, Kunming 650091, China; 3. School of Chemistry and Biotechnology, Yunnan University of Nationalities, Kunming 650031, China

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摘 要 采用热重分析-单滴微萃取-气相色谱-质谱(TG-SDME-GC-MS)联用系统和傅里叶变换红外光谱,研究了咖啡酸的热解行为。设定热重分析仪5 ℃/min的升温速率及400 mL/min的氮气流量,在160～360 ℃温度范围内,采用乙醇对热解逸出物质进行单滴微萃取,然后利用GC-MS分离分析,监测了咖啡酸5种主要热解逸出产物相对含量随温度升高的动态变化情况。使用傅里叶变换红外光谱分析了咖啡酸所对应各失重点固体剩余物的特征官能团变化情况。结果表明,咖啡酸热失重的主要原因是在240～360 ℃产生大量的邻苯二酚,在200～220 ℃热解产生4-乙基邻苯二酚。另外,咖啡酸在230 ℃下已完全裂解。该方法的建立为温度连续上升模式下的物质热解行为分析提供了借鉴和参考。

关键词： 热重分析 单滴微萃取 气相色谱-质谱 傅里叶变换红外光谱 热分解 逸出组分 咖啡酸

Abstract： The thermal decomposition behavior of caffeic acid was investigated using the thermogravimetry-single drop microextraction-gas chromatography-mass spectrometry (TG-SDME-GC-MS) and Fourier transform infrared spectroscopy. The heating rate and the air flow rate were set at 5 ℃/min and 400 mL/min, respectively. The evolved components of caffeic acid from thermogravimetry were extracted with ethanol by single drop microextraction in the temperature range of 160~360 ℃. Then the extract was separated and analyzed by GC-MS. Thus, the dynamic changes of the relative contents of 5 main pyrolysis products with the increase of temperature were identified and monitored. The alterations of functional groups of the solid residues at each weight-lose-point were analyzed by Fourier transform infrared spectroscopy. The results showed that the main reason for the weight loss of caffeic acid can be concluded to the generation of mass pyrocatechol at 240~360 ℃ and 4-ethylcatechol at 200~220 ℃. The molecular structure of caffeic acid was completely decomposed at 230 ℃. This method provides a significant approach for the investigation of the thermal decomposition behavior of material with the continuous rising of temperature.

Keywords： thermogravimetric analysis single drop microextraction gas chromatography-mass spectrometry (GC-MS) Fourier transform infrared spectroscopy thermal decomposition evolved species caffeic acid

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Corresponding Authors: 杨柳,博士,高级工程师,从事烟草化学研究. Tel: (0877)2968271, E-mail: liuyang929@126.com. Email: liuyang929@126.com

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