EXTRACT OF *ALBIZIA KALKORA* LEAVES

by

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In this paper, a mixture of methanol, ethanol, benzene and ethanol were used to extract the leaves of Albizia kalkora (Roxb.) Prain. Then, using Fourier transform infrared (FT-IR) spectroscopy, gas chromatography-mass spectrometry (GC-MS), pyrolysis as chromatography - mass spectrometry, and thermogravimetric analysis (TGA) and other advanced instruments Albizia kalkoraleaves were analyzed to determine their composition. According to the test data, Albizia kalkoraleaves contain epinephrine, trans squalene, stachydrine and other chemical components with high medicinal value, indicating that Albizia kalkoraleaves have potential applications in the pharmaceutical industry. This study has expanded the utilization value of Albizia kalkoratrees and provided technical support for the resource utilization of Albizia kalkoratrees.

Key words: *Albizia kalkora, GC-MS, TGA, Py-GC-MS, medicinal value*

Introduction

With the speed development of the economy and improvements living standards, society has more requirements for health, and the pharmaceutical industry is developing faster and faster. The pharmaceutical industry also has an increasing demand for raw materials. As one of the main sources of the pharmaceutical industry [1-3], the research and development of different plant medicines are becoming more important. *Albizia kalkora (Roxb.) Prain* is a small deciduous tree, usually 3-8 meters high, with dark brown branches and conspicuous lenticels. *Albizia kalkoratrees* are widely distributed in across China, Vietnam, Myanmar, and India. They are physically found in hillside thickets and sparse forests. Its main values are that the species is fast growing and capable of withstanding drought and poor soil quality. The bark is resistant to moisture, and the flowers are colorful. Although *Albizia kalkoratrees* can be used as ornamental plants and have certain economic value, some of their chemical components can be used as raw materials and intermediate products of other products for further resource utilization.

In this paper, different reagents, such as mixtures of ethanol, methanol, benzene and ethanol were used to extract leaf samples of *Albizia kalkora (Roxb.) Prain*. The FT-IR, GC-MS, Py-GC-MS, TD-GC-MS, TGA and other analytical and detection technologies were used to

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analyze and detect the components of the samples. Through testing, it was found that the leaves of *Albizia kalkoratree* contain a large number of chemical components, such as squalene [4-6], Adrenaline I [7], Phytol, 9-Octadecenamide, and (Z)-, 9,12-Octadecadienoic acid (Z, Z)-, which can be used as pharmaceutical ingredients, solvents, and raw materials for daily necessities, expanding the utilization value of the *Albizia kalkoratree*.

Materials and experimental methods

Materials

We were collected fresh plants of *Albizia kalkoratree* from the Luoyang forest area of the Henan province, China, and the leaves were preliminarily polished in the laboratory and crushed to 40-60 mesh for reserve. Methanol (analytically pure) and ethanol (analytically pure) were purchased from Tian in Fuyu Fine Chemical Co., Ltd., and benzene (analytically pure) was purchased from Yantai Shuangshuang Chemical Co., Ltd. Deionized water was used from the laboratory.

Sample preparation

The 10 g of the sample and 300 mL of the reagent were placed in a distillation flask for mixing. After boiling, and the solution was removed, cooled, filtered, and steamed to 10 mL for later use.

Determination of composition

The FT-IR analysis

Potassium bromide was ground into fine powder, mixed with a few samples of *Albizia kalkora* leaves, crushed and placed into infrared spectrometer for analysis [8-10].

The GC-MS analysis

The sample was added to the instrument, and following procedure was utilized: the high purity helium was used a carrier gas at a flow rate of 1 mL/min and the separation ratio was 50:1. The gas chromatographic temperature procedure started at 50 °C, and then we went up to 250 °C at a rate of 10 °C/min. Finally, we went up to 280 °C at a rate of 5 °C/min [11-13].

The Py-GC-MS analysis

We analyzed the powder by pyrolysis gas chromatography-mass spectrometry. Powder samples were placed in the instrument, and we used high-purity helium gas as the carrier gas. The following procedure was utilized: pyrolysis temperature was 850 °C, pyrolysis time was 30 seconds. We set the initial temperature of Py-GC at 40 °C for 2 minutes. Next, we will increase the temperature to 120 °C at the rate of 5 °C/min. Finally, we increased the temperature to 200 °C at a rate of 10 °C/min for 2 minutes [14-16].

The TD-GC-MS analysis

The powder samples were placed into the injection port of the instrument, and column temperature procedure was set as: The initial temperature is 30 ℃, and then we increase the temperature to 100 ℃ at the rate of 10 ℃/min. We then raised the temperature to 250 ℃ at a rate of 8 ℃/min. We then raised the temperature to 280 ℃ at the rate of 5 ℃/min. Finally, hold at 280 ℃ for 2 minutes [17-22].

The TGA analysis

The leaf samples were analyzed by a thermogravimetric analyzer. We used nitrogen as the equilibrium gas, and the TGA temperature program was set from 30-850 °C at a rate of 15 °C/min and 30 °C/min, respectively, and, finally, the temperature was maintained at 850 °C for 5 minute [23-25] in both procedures.

Results and discussion

The FT-IR analysis results

First, FT-IR was used to analyze the sample materials, as shown in fig. 1.

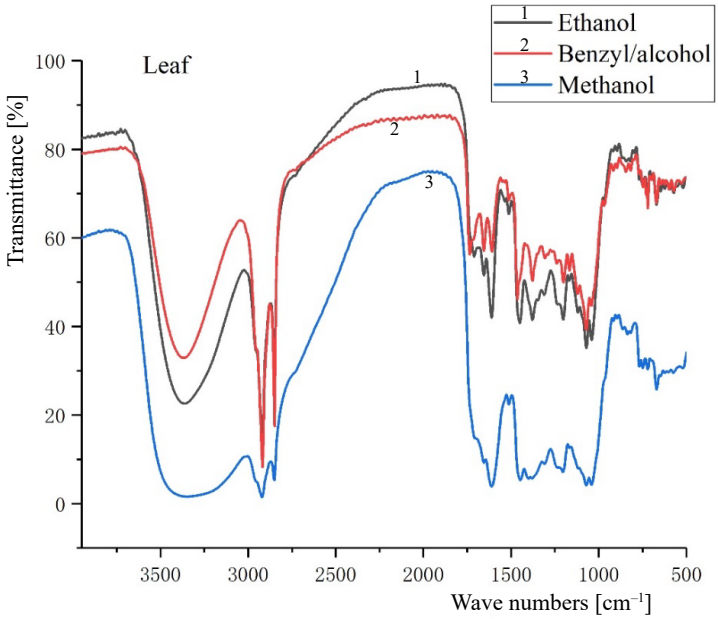

Figure 1. Infrared spectra of extracts of *Albizia kalkora* leaves

The FT-IR test results showed a similar peak for all three samples at 3500-3250 cm^{-1}, indicating that there was a -OH bond in the extracts of the leaves of the three species of *Albizia kalkora (Roxb.) Prain*. The wave peaks were strong and wide, indicating that the extracts might contain alcohols and phenols. The wave peak appearing at the wave number range of 3000-2750 cm^{-1} indicated that there was a C-H bond in the extract of *alacia alba*, with strong and sharp wave peak absorption intensity and double peaks, indicating that the extract may contain alkane compounds. We found that the peaks appeared at wave Numbers of 1650, 1500-1300 and 1200-1000 cm^{-1}. It indicated the presence of C-O, C-C, C=O and C=C bonds in the extract of *alacia* bark leaves, which represent aldehydes, ketones, ethers, acids, esters, and olefin compounds.

The GC-MS analysis results

The GC-MS test results of the three reagents extracted from the leaves of *Albizia kalkora* are shown in figs. 2-4.

According to the GC-MS test results in fig. 2, the main components of the ethanol extracts from the leaves of *Albizia kalkora* are Myo-Inositol, 4-C-methyl- (77.28%),

9-Octadecenamide, (Z)- (4.47%), Squalene (3.41%), butyl 9-tetradecenoate (2.08%), l-(+)-Ascorbic acid 2,6-dihexadecanoate (1.80%), Phytol (1.40%), 1,2,3-Benzenetriol (0.35%), and 9,12-Octadecadienoic acid (Z,Z)- (0.265%).

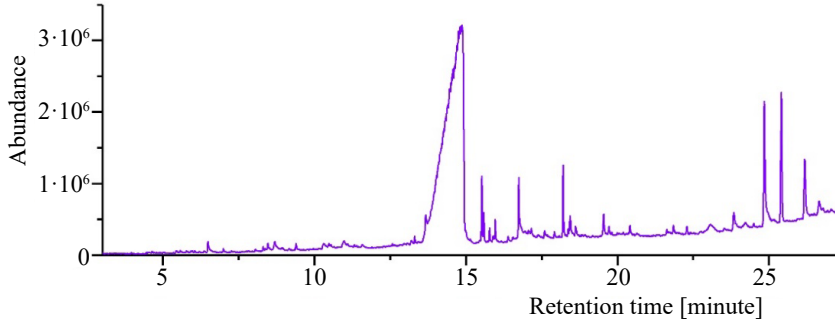

Figure 2. The GC-MS ion spectra of ethanol extract from *Albizia kalkora* leaves

According to the GC-MS test results in fig. 3, the main components of the benzene and ethanol extracts from *Albizia kalkora* leaves are Myo-Inositol, 4-C-methyl- (74.98%), Squalene (5.15%), 9-Octadecenamide, (Z)- (2.64%), Phytol (1.44%), dibutyl phthalate (0.94%), and 9,12,15-Octadecatrienoic acid, (Z,Z,Z)- (0.51%).

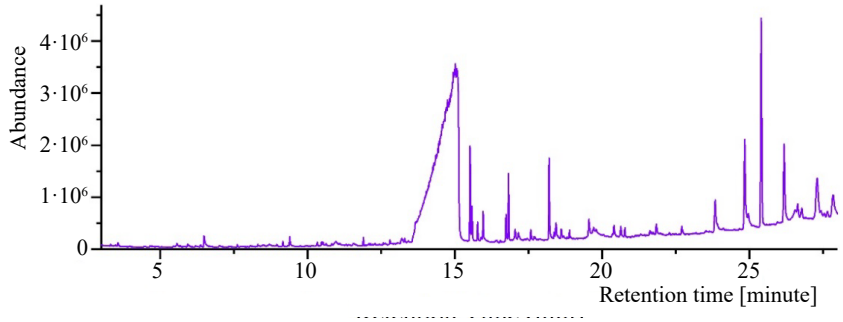

Figure 3. The GC-MS ion spectra of benzene-ethanol extracts from *Albizia kalkora (Roxb.) Prain*

As we can see from the GC-MS test results in fig. 4, methanol extract from *Albizia kalkora* leaves mainly contains the following components: Myo-Inositol, 4-C-methyl- (24.97%), Squalene (11.65%), 13-Docosenamide, (Z)- (7.04%), Benzoic acid, 3,4,5-trihydroxy-, methyl ester (6.19%), Phytol (4.31%), and 1,2,3-Benzenetriol (1.95%).

From figs. 2-4, it can be seen that the leaves of *Albizia kalkora* have the following substances with higher utilization value:

- Squalene -oral administration of Squalene can treat high and low blood pressure, diabetes, cirrhosis, cancer, and tooth decay. External applications of Squalene can treat tonsillitis, bronchitis, tuberculosis, rhinitis, gastric ulcer, gallbladder and bladder stones, rheumatism, and neuralgia.
- Phytol - it is regarded as the basic material to produce vitamins K1 and E.
- The 9,12-Octadecadienoic acid (Z, Z)-, it primarily used as a raw material for paints and inks. It can also be used to produce polymers such as polyamide, polyester and polyurea. It can be used as the raw material for the treatment of atherosclerosis drugs, which have positive treatment effects on cardiovascular and cerebrovascular diseases.

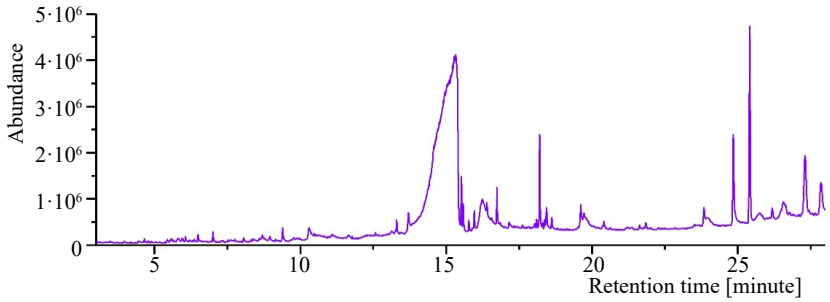

Figure 4. The GC-MS ion spectra of methanol extract from *Albizia kalkora* leaves

The Py-GC-MS analysis results

The Py-GC-MS test results of *Albizia kalkora* leaves are shown in fig. 5.

As we can see from the Py-GC-MS test results in fig. 5, the main components of *Albizia kalkora* leaves are Cyclopropyl carbinol (6.52%), Phenol, 3-methyl- (2.87%), *o*-Xylene (2.29%), Acetyl bromide (1.99%), *n*-Hexadecanoic acid (1.61%), 5-Chloro-1-pentyne (1.59%), 1-Hexadecanol, 2-methyl- (1.59%), Indene (1.42%), Boron, trihydro(pyridine)-, (T-4)- (1.36%), Phenol (1.02%), Falcarinol (0.79%), and 1,4-Benzenediol, 2,3,5-trimethyl- (0.20%).

Figure 5 shows that the leaves of *Albizia kalkora* have the following substances with higher utilization value:

- Cyclopropyl carbinol -Cyclopropyl carbinol is an organic synthesis intermediate and pharmaceutical intermediate, which can be applied to laboratory research and development and chemical and pharmaceutical synthesis processes.
- The *o*-Xylene- *O*-xylene is used primarily as a chemical raw material and solvent. It can also be used in the production of pesticides and drugs, and as an additive in aviation gasoline.
- Phenol - phenol is an important organic synthetic raw material, which can be used to manufacture phenolic resin, phenolphthalein, salicylic acid, alkyl phenol and other chemicals, such as solvents.
- Falcarinol -Falcarinol has anti-tumor, antibacterial and neuroprotective effects.
- The 1,4-Benzenediol, 2,3,5-trimethyl- - It is the main ring of vitamin E and can be condensed with different plant alcohols to obtain vitamin E.
- Phenprobamate - It can be used as a relaxant for skeletal muscle. It can be used for lumbago, limb pain, muscle pain after exercise, rheumatoid arthritis, and other muscle issues.

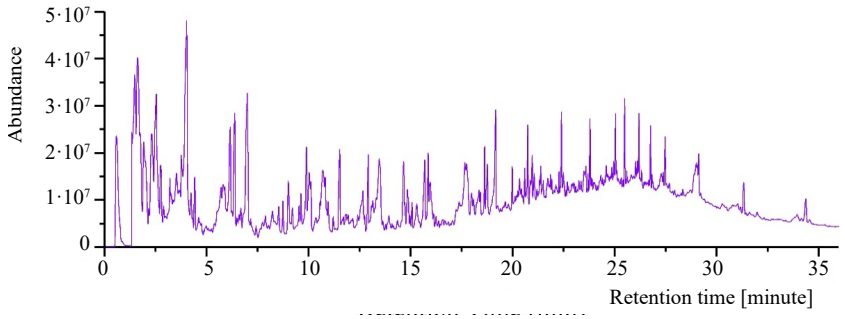

Figure 5. Total ion content of *Albizia kalkora* leaves by Py-GC-MS

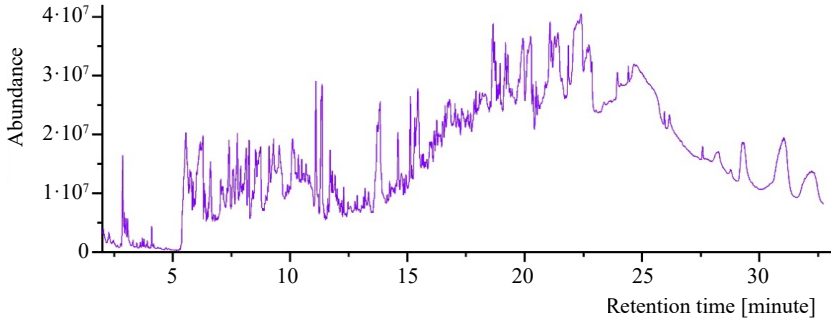

Figure 6. Total ion content of *Albizia kalkora* leaves by TD-GC-MS

The TD-GC-MS analysis results

The TD-GC-MS test results of *Albizia kalkora* leaves are shown in fig. 6 [26].

As we can see from the TD-GC-MS test results in fig. 6, the main components of *Albizia kalkora* leaves are *n*-Hexadecanoic acid (5.25%), acetic acid (5.15%), 2-Propenoic acid (1.13%), 9,12-Octadecadienoic acid (Z,Z)- (12.06%), 2-Propenoic acid, 2-methyl- (5.19%), 4-Hydroxy-3-methylacetophenone (2.66%), pyridine (1.25%), 1,2,6-Hexanetriol (1.20%), Cyclohexasiloxane, dodecamethyl- (1.12%), 2-Butenoic acid, (E)- (0.94%).

Figure 6 shows that the leaves of *Albizia kalkora* have the following substances with higher utilization value:

– The 9,12-Octadecadienoic acid (Z,Z)- - It can be used as a raw material in paints and inks and can also be used to produce polymers such as polyamide, polyester and polyurea.
– The n-Hexadecanoic acid - It can be used in the production of daily necessities, such as candles, soap, grease, and softeners, and as a standard substance for gas phase analysis.
– The 2-Propenoic acid, 2-methyl- - It is an important organic chemical raw material and polymer intermediate in the manufacturing of coatings, adhesives and ion-exchange resins.
– Acetic acid - acetic acid is primarily used for the preparation of ethylene acetate, acetic ester, and chloroacetic acid. It can also be used as a solvent.
– Pyridine - In addition to being used as a solvent, pyridine is also used in the industry as a dye aid and as a starting material of a range of products, including pharmaceuticals, dyes, adhesives, and explosives.
– The 1,2,6-Hexanetriol - It can be used as a tobacco wetting agent, solvent, coupling agent for special hydraulic fluids, and a raw material in synthetic rubber.

The TGA results

The TGA test results of *Albizia kalkora* leaves are shown in fig. 7.

The test results showed that the leaves of *Albizia kalkora* went through three stages of weight loss. The two heating modes showed basically the same form. In the first stage, the samples lost weight at 100 °C, indicating that this stage is water loss, primarily from the loss of free water and binding water. In the second stage, weight loss occurs between 300-400 °C, which indicates that this stage is the thermal weight loss stage, primarily attributed to the decomposition of lignin and other components. The Weightlessness rate in this stage reaches a maximum value, which is the main stage of pyrolysis. In the third stage, weight loss occurs between 600-800 °C, indicating that this stage is the carbonization stage. After this, the weight of the sample does not change significantly.

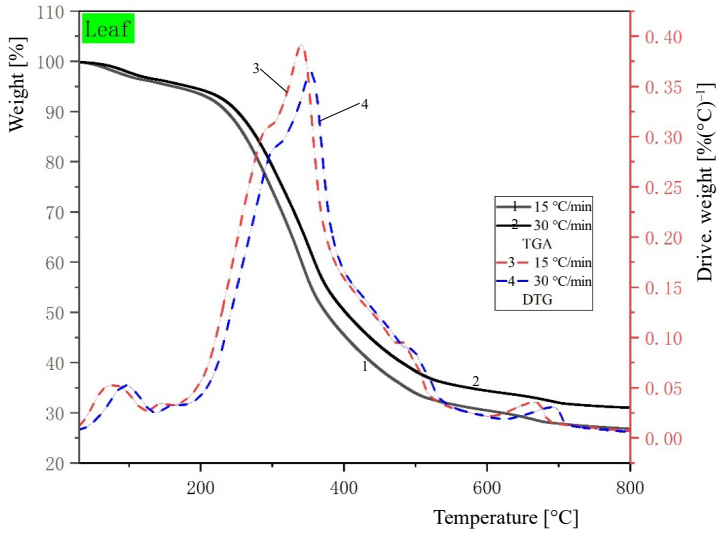

Figure 7. The TG chart of different heating rates of *Albizia kalkora* leaves

Conclusion

It can be seen from the above results that the extracts of the leaves of *Albizia kalkora* contain a large number of chemical components, such as Adrenaline, Squalene and Stachydrine, which are of high medicinal value and can be used as raw materials for the pharmaceutical industry. Another example is n-hexadecanoic acid, 9, 12-octadecadienoic acid (Z,Z)-, 2-methyl-, an important intermediate in organic chemical raw materials and polymers, which can be used to make chemical products such as paints, candles, soap, and coatings. Therefore, *Albizia kalkora*leaves can be used as a new material in the pharmaceutical and chemical industries and agricultural production, which has high potential application value. This experiment provides a theoretical basis for the further development and utilization of *Albizia kalkora*leaves and promotes the utilization of biological resources.

References

[1] Shallan, A, I., Priest, C., Microfluidic Process Intensification for Synthesis and Formulation in the Pharmaceutical Industry, *Chemical Engineering and Processing - Process Intensification*, *142* (2019), Aug., 107559
[2] Thomas, F., *et al.,* The Impact of Quality Culture on Operational Performance - An Empirical Study from the Pharmaceutical Industry, *PDA Journal of Pharmaceutical Science and Technology*, *72* (2018), 5, pp. 531-542
[3] Kumar, A., *et al.,* When Risks Need Attention: Adoption of Green Supply Chain Initiatives in the Pharmaceutical Industry, *International Journal of Production Research, 57* (2019), 11, pp. 3554-3576
[4] Chen, G., *et al.,* Cloning and Analyzing a Squalene Synthase Gene in Pseudostellaria Heterophylla, *Gene Reports, 12* (2018), Sept., pp. 39-46
[5] Bonafonte, J. M. L., *et al.,* Current Insights into the Biological Action of Squalene, *Molecular Nutrition & Food Research, 62* (2018), 15, e1800136
[6] Qian, J. Y., *et al.,* Positive Selection of Squalene Synthase in Cucurbitaceae Plants, *International Journal of Genomics*, *2019* (2019), ID 5913491
[7] Maselli-Campagna, G., *et al.,* Research on Acute Pulmonary Edema Induced with Adrenaline, *I. Study of the Protective Action of Butydrine. Bollettino Della Società Italiana Di Biologia Sperimentale, 45* (1969), 15,

[8] Wu, Y., Wang, Y., An FTIR Study of Kyanite in the Maobei Kyanite-Bearing Eclogites from the Sulu Orogenic Belt, Eastern China, *Journal of Earth Science, 29* (2018), 01, pp. 21-29
[9] Bobrowski, A., *et al.,* Studies on Thermal Decomposition of Phenol Binder Using TG/DTG/DTA and FTIR-DRIFTS Techniques in Temperature Range 20-500 °C, *China Foundry, 15* (2018), 02, pp. 145-151
[10] Ren, X., *et al.,* TG-FTIR Study on the Pyrolysis Properties of Lignin from Different Kinds of Woody Biomass, *Paper and Biomaterials, 3* (2018), 02, pp. 1-7
[11] Tu, H., *et al.,* Analysis of Low Melting Point Fat Constituents of Black Soybeans by HS-SPME-GC-MS, *Medicinal Plant, 8* (2017), 05, pp. 4-5
[12] Tahghighi, A., *et al.,* GC-MS Analysis and Anti-Mosquito Activities of Juniperus Virginiana Essential Oil Against Anopheles Stephensi (Diptera: Culicidae), *Asian Pacific Journal of Tropical Biomedicine, 9* (2019), 04, pp. 168-175
[13] Blanchard-Wrigglesworth, E., Bushuk, M., Robustness of Arctic Sea-Ice Predictability in GCMs, *Climate Dynamics, 52* (2019), 19, pp. 5555-5566
[14] Andersone, I., *et al.,* A Study of Thermo-Hydro-Treated (THT) Birch Wood by Chemical Analysis and Py-GC/MS, *Holzforschung, 73* (2019), 7, pp. 653-661
[15] Cai, W., *et al.,* Py-GC/MS Analysis on Product Distribution of Two-Staged Biomass Pyrolysis, *Journal of Analytical and Applied Pyrolysis, 138* (2018), Mar., pp. 62-69
[16] Ma, Z., *et al.,* Pyrolysis Behaviors of Oilfield Sludge Based on Py-GC/MS and DAEM Kinetics Analysis, *Journal of the Energy Institute, 92* (2019), 4, pp. 1053-1063
[17] Yan, P., Analysis on Biohealth Function of Dried Pinus Biomass by TD-GC-MS: Analysis on Biohealth Function of Dried Pinus Biomass by TD-GC-MS, Shenzhen, China, 2011
[18] Peng, W. X., Analysis on Health Risk of Volatiles of Pinus Massoniana Biomass by TD-GC-MS: Analysis on Health Risk of Volatiles of Pinus Massoniana Biomass by TD-GC-MS, Shenzhen, China, 2011
[19] Wu, Q. D., *et al.,* Analysis on Volatiles Release Behavior of Poplar Wood Powder at Different Temperature by TD-GC/MS and Environmental Protection Evaluation, *Chinese Forestry Science and Technology, 11* (2012), 03, pp. 79
[20] Gallego, E., *et al.,* Development of a Method for Determination of VOCs (Including Methylsiloxanes) in Biogas by TD-GC/MS Analysis Using Supel™ Inert Film Bags and Multisorbent Bed Tubes, *International Journal of Environmental Analytical Chemistry, 95* (2015), 4, pp. 291-311
[21] Akyildiz, F. T., Vajravelu, K., Galerkin-chebyshev Pseudo Spectral Method and a Split Step New Approach for a Class of Two Dimensional Semi-linear Parabolic Equations of Second Order, *Applied Mathematics & Nonlinear Sciences, 3* (2018), 1, pp. 255-264
[22] Caraballo, T., *et al.,* An Iterative Method for Non-Autonomous Nonlocal Reaction-Diffusion Equations, *Applied Mathematics & Nonlinear Sciences, 2* (2017), 1, pp. 73-82
[23] Guo, G., *et al.,* Characterization of Moisture Mobility and Diffusion in Fresh Tobacco Leaves during Drying by the TG-NMR Analysis, *Journal of Thermal Analysis and Calorimetry, 135* (2019), 4, pp. 2419-2427
[24] Zhao, X., *et al.,* Evolved Gas Analysis of PEP-SET Sand by TG and FTIR, *Journal of Analytical and Applied Pyrolysis, 127* (2017), Sept., pp. 490-495
[25] Zhu, Y., *et al.,* Pyrolysis Study of Huainan Coal with Different Particle Sizes Using TG Analysis and Online Py-PI-TOF MS, *Journal of the Energy Institute, 93* (2019), 1, pp. 405-414
[26] Peng, W. X., *et al.,* TD-GC-MS Analysis on Thermal Release Behavior of Poplar Composite Biomaterial under High Temperature, *Journal of Computational and Theoretical Nanoscience, 9* (2012), 9, pp. 1431-1433

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