Studies on extraction of concrete from flowers of Sophora Japonica.L by supercritical carbon dioxide

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Abstract: The experimental research of the concrete extraction from flowers of Sophora Japonica. L. by means of supercritical carbon dioxide (SC-CO_2) as a solvent was studied. The apparatus with extractor volume in 1L was setup. Experiments were conducted at pressures between 10 MPa and 30 MPa and temperatures between 313K and 333K. The purity of carbon dioxide reached 99%. Flowers of Sophora Japonica.L, which were collected in May this year in Dalian, China, had fresh samples and dry samples. The experiments show that concrete can be extracted with supercritical carbon dioxide from fresh and dry flowers of Sophora Japonica.L. The concrete extracted were ropy yellow and had a strong odor of flowers of Sophora Japonica.L. The chemical composition of the extracts was primarily determined using GC-MS analysis methods. The major compounds of the essential oil were diethyl phthalate, rutinoside, isorhamnetin, hexadecenoic acid, tetradecadecenoic acid, quercetin, eicosanoic acid, hexadecanoic acid, octadecadienoic acid and so on. Experiments turned out that both the yield of concrete obtained from fresh flowers and that from dry flowers was nearly 1% (wt%).

This article laid emphasis on improved methods of extraction process in order to increase the yield of concrete. Flowers of Sophora Japonica.L were dealt with by swelling with an alcohol solution of certain concentration (mass of solute: mass of solvent=1:3, at room temperature 20? , 15min). The results showed that the yield obtained from flowers with swelling treated was more than 10 times than that obtained from flowers without swelling treated.

Keywords: Sophora Japonica.L., concrete, supercritical carbon dioxide extraction, swelling treatment.

1. Introduction

Sophora Japonica. L. belongs to the family Leguminosae and is widely distributed in China, especially in the Loess Tableland and the Huabei Plain. Its flowers, Flos Sophorae, are very fragrant and a favorite with people. Every year Flos Sophorae Festival is held when Flos Sophorae is blossoming in Dalian. But after the florescence of Flos Sophorae, snowwhite petals fell become to useless litter in cities. If we can extract flavor from Flos Sophorae, we not only make full use of natural resource, but also avoid pollution. Especially Flos Sophorae extracts can be used in the cosmetology industry to impart flavor and nutrition to a number of personal care products. So we first used supercritical CO2 to obtain Flos Sophorae extracts.

The objectives of the work were to study the influence of fresh samples (F), and dry samples (D) on the yield, temperature (T), and pressure (P) on the yield, flowers with swelling treated (S), and flowers without swelling treated (N) on the yield and composition of the extract. The

extractions were performed at pressures of 10 and 30MPa, and temperatures from 313 to 333 K.

2. Material and methods

2. 1. Raw material

Flowers of Sophora Japonica.L, which were collected in May this year in Dalian, China, had fresh samples and dry samples. Dry samples were stored at 60°C for a day prior to its usage.

2. 2. The apparatus

The apparatus with extractor volume in 1L was setup. The purity of carbon dioxide reached 99%. Carbon dioxide gas at a pressure of 5 MPa was compressed by piston pump. A stainless steel extraction column (E) was placed in the water bath. Supercritical CO2 was expanded across the first micro-metering valve (MV1). Supercritical CO2 with Flos Sophorae extracts from the MV1 was delivered to the first receiver column (R1), the second micro-metering valve (MV2), the second receiver column (R2) and the third micro-metering valve (MV3) connected to a rotameter (RM) one by one. A gas flowmeter (FM) was connected after the rotameter for the measurement of the amount of CO2 consumed during the process. Each receiver had a micro-metering valve, which was used to let extracts off, at their bottoms. Solute (Flos Sophorae extracts) was collected in a test tube every 10 min and the flow rate was monitored.

2. 3. Results and discussion

Table 1: experimental results
(3,4,9,13, for dry samples runs in blue; the others for fresh samples runs in black)

No.	Pressure/ MPa	Temperature/?	Yield /%
1	15	35	0.143
2	30		0.500
3	30		0.250
4	30	40	0.625
5	30		1.143
6	25		0.143
7	20		0.860
8	10		0.000
9	28	50	0.261
10	25		1.538
11	22		0.464
12	15		2.072
13	15		1.625
14	25	60	7.907
15	15		4.444
16	10		0.000

Table 1 shows the yield of concrete obtained from fresh flowers and that from dry flowers at different condition of pressure and temperature. All products were yellow concrete. Based on these data, Fig.1 and 2 were drawn to describe the relationships between operating conditions and the concrete yield.

From Fig.1, we can find out that an increase in the yield of fresh flowers concrete as the pressure increased from 15 to 30 MPa at the temperature of 35? . The yield curves of concrete extracted at the temperature of 40? and 50? were changed for no polar substance is not suitable for extraction at high pressure. The extraction yield at 30MPa was high because the extract contained impurity. The two curves of 40? and 50? were very similar, but the curve of 50? was on the left and top to the curve of 40? . Maybe the increase of temperature made up for the decrease of pressure and the density of supercritical CO2 achieved the same. The yield curve at 60? was not like the curves at 35? 40? 50? and went up directly. Because the extract at 60? had undesirable substance and was different from that at 35? 40? 50? . The yield curve of dry samples concrete was very different from that of fresh samples concrete. The yield curve decreased as the pressure increased from 15 to 27 MPa at the temperature of 35? . Because dry flowers extraction data are few, we should continue studying dry samples extraction.

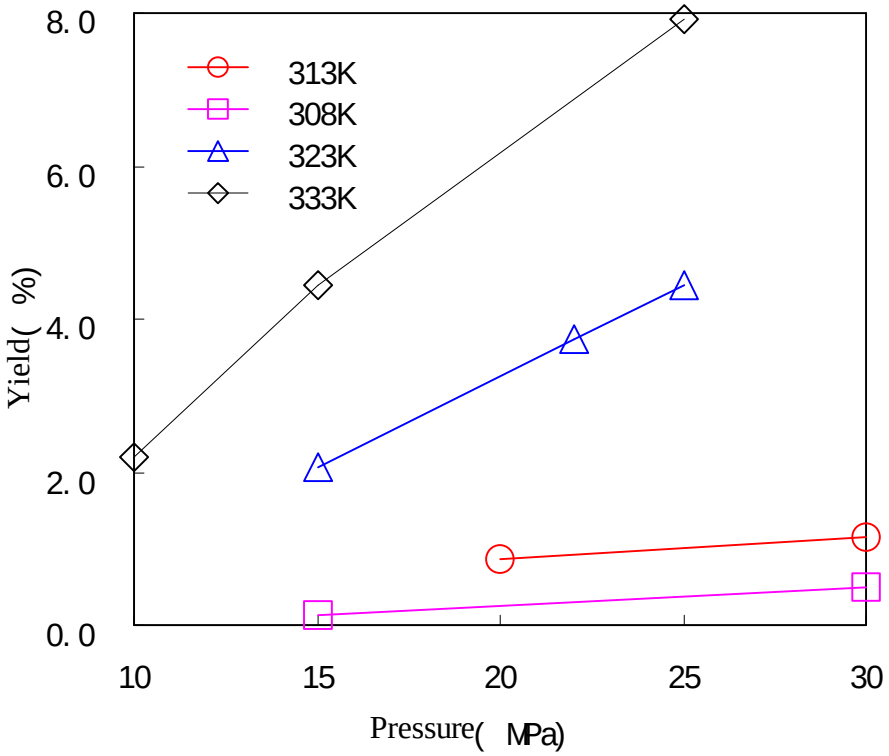

Fig.1 Relationships between pressure and yield at constant temperatures

From Fig.2, it described the extraction yield curves of fresh and dry flowers at the same pressure and different temperature. The yield curves of fresh and dry flowers at any pressure increased as the temperature increased. The rule of fresh flowers yield curves at 15 and 25MPa was separated into two parts for the two curves were crossed. When the temperature

was under 52? , for example, the extraction yield at 15MPa and 40? was bigger than the yield at 25MPa and the same temperature, and was smaller than the yield at 30MPa and the same temperature; While the temperature was above 52? , for example, the extraction yield at 25MPa and 60? was as double as yield at 30MPa and the same temperature. Maybe there is a minimum of fresh flowers concrete yield between 15 and 22MPa. Compared with dry flowers concrete yield, the fresh flowers concrete yield was always larger than it. Because fresh flowers had moisture, products carried some moisture sothat the apparent yield was larger. We could see that dry flowers concrete yield at 15MPa and 50? was larger than fresh flowers concrete yield at the pressure of 22 and 25 MPa and the same temperature. We think that mass transfer coefficient became larger after fresh flowers were removed moisture, and that low pressure was more suitable for flavor extraction. This is a so good operation condition that dry flowers are extracted at 15MPa and 50? that operation pressure is low, operation cost is reduced, and the storage of fresh flowers is solved.

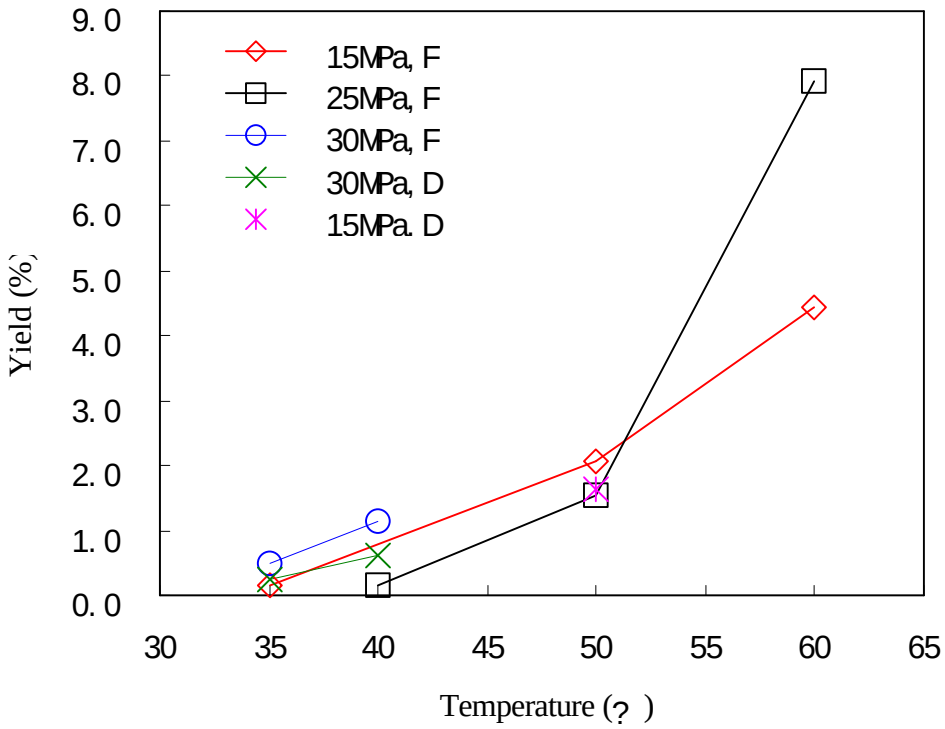

Fig.2 Relationships between temperature and yield at constant pressures

Composition analysis was determined using a GC-MS. The compositions of essential oil isolated from concrete are as follows: diethyl phthalate 19.84 wt%, rutinoside 7.22 wt%, isorhamnetin 1.88 wt%., hexadecenoic acid 1.39 wt%, tetradecadecenoic acid 1.39 wt%, quercetin 2.87 wt%, eicosanoic acid 0.34 wt%, hexadecanoic acid 1.9 wt%, octadecadienoic acid 2.57 wt%. Fig.3 is the GC-MS analysis diagram of the essential oil composition.

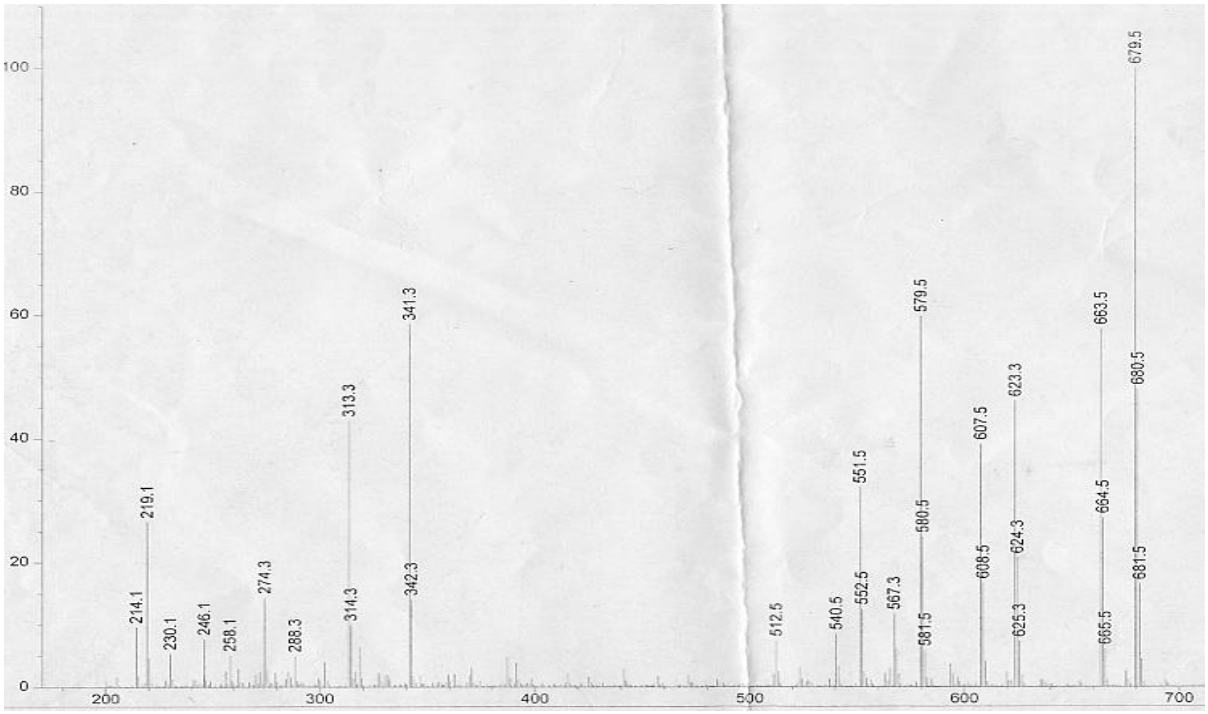

Fig.3 GC-MS diagram

Table 3: experimental data about samples with swelling treated

No.	P MPa	T ?	Yield %	Pre-treatment	Properties
16	27	50	1.010	Without-	yellow concrete
17	27	50	1.580	20 mesh	yellow concrete
18	27	50	4.770	H_2O + alcohol	yellow ropy concrete
19	27	50	16.48	Added alcohol	yellow ropy concrete
20	27	40	14.38	Added alcohol	yellow ropy concrete
21	20	50	4.850	Added alcohol	pink liquid + yellow ropy concrete
22	27	50	83.33	60 mesh, added alcohol	Green concrete

Table 3 showed the extraction data of dry flowers with swelling treated and without swelling treated. From the comparison of experiment 16 and 17, we could see that the concrete yield of cracked samples was one and a half as large as the yield of noncracked samples. So cracking samples is benefit for mass transfer. The yield of experiments 18 ,19 and 20 is 5~15 times as large as the yield of experiment 16. So it is very effective to improve the concrete yield that flowers of Sophora Japonica.L are dealt with swelling with an alcohol solution of certain concentration (mass of solute: mass of solvent=1:3, at room temperature 20 ? , 15min). Experiments 19 and 20 indicated that the concrete yields of samples with the same treatment at 40? and 50? was near. But experiment 21 showed that the concrete yield decreased prominently as pressure decreased, for example, the concrete yield at 20MPa

and 50？ was 1/4 as large as that at 27MPa and the same temperature. The curve for experiment 22 was suddenly risen up, maybe due to the composition of extracts was different.

3.Conclusion

This is the first study about flavor extraction of Flos Sophorae using supercritical CO2. The work has a much of disadvantages to resolve in the future. But it is clear that Flos Sophorae flavor can be extracted using supercritical CO2, and that low pressure is suitable for the concrete yield, and that it can effectively improve the concrete yield that flowers of Sophora Japonica.L that swelling dry flowers with an alcohol solution of certain concentration (mass of solute: mass of solvent=1:3, at room temperature 20？, 15min).