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Fatty and organic acids, and barbaloin in various parts of Aloe species dried at different drying temperatures#

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Abstract : The fatty and organic acids, and barbaloin in various parts of aloe species dried at different temperatures were analyzed by GC and HPLC. Seven fatty acids and six organic acids were identified and quantified. In the case of fatty acids, generally, the contents of palmitic and eicosanoic acid were abundants, and compared to the total contents of seven fatty acids, *Aloe arborescence* variant 1 was abundant, but *Aloe saponaria* was poor. And six fatty acids were distributed in the aloe species with the exception of linoleic acid. The contents of malic, citric and oxalic acids in the aloe species were higher than those of other acids, and compared to the sum of contents of six organic acids, *Aloe saponaria* was high, but *Aloe arborescence* variant 1 was low. Therefore *Aloe arborescence* variant 1 was abundant in total fatty acids, but poor in total organic acids. The contents of fatty and organic acids in the sample dried at 65℃ and 80℃ air circulation were almost similar. The contents of fatty and organic acids in the freeze-dried samples were lower than in the other dried samples.

The contents of barbaloin in *Aloe arborescence* and *A. arborescence* variant 1 were higher than those of other various samples, and barbaloin was not detected in *Aloe saponaria*. As the drying temperature was increased, the contents of barbaloin in the various parts of the *Aloe vera* decreased (Received April 21, 1993; accepted June 8, 1993).

Aloe species are useful plants used multipurposely by human beings. The leaves of aloe species are well-known crude drugs having peptic and laxative activities. The aloe leaves have been used as a folk drug for many purposes and as materials of some cosmetic and health foods.[14,20] The plants of aloe species have been widely used as remedies for burns, insect bites and gastro-intestinal disorders.[14] Fly and Kiem[16] carried out an investigation to ascertain whether *Aloe vera* exhibits antimicrobial activity. Barbaloin was the main laxative component in aloe species, an anthraquinone glycoside.[20] But until now researches related to useful effective components in the above stated plants were lacking. Especially, the study of the plant cultivated in regions in Korea was very little. Therefore, the fatty and organic acids, and barbaloin in the various parts of aloe species dried at different temperature were compared to study the relation to cosmetic, health foods and medicines.

Materials and Methods

The leaves of *Aloe vera, A. saponaria, A. arborescence* and *A. arborescence* variant 1, 2, which had been culti-

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vated for five years in the field of Yousung Aloe Farm located in Taejon, were collected and separated into skin, gel and whole leaf parts. The samples were dried at 65℃ and 80℃ with air ciculation, and freeze-drying was carried out only for whole leaves. The dried matter was made to pass 60 mesh sieve, and the pulverized samples were used to analyze fatty acids, organic acids and barbaloin. Physico-chemical characteristics of the soil were analyzed to know the level of fertility and is shown in Table 1.

Some properties of the field soils were analyzed by R.D.A. standard method.[1] Organic matter, total nitrogen, and available phosphorus were determined by Tyurin, Kjeldahl and Truog methods, respectively. Exchangeable potassium and sodium were analyzed by flame photometry. Magnesium and calcium were detected by atomic absorption spectrophotometry, respectively. Cation exchange capacity (CEC) was determinded by 1 N-ammonium acetate method.

Fatty and organic acids

For methyl esterification of the acids, 10 g of dried samples was esterified by 60 m*l* of H_2SO_4+MeOH (12 % v/v) mixture and shaken for twenty hours. The concentration of the glutaric methyl alcohol was made to 1.25 mg/m*l* as an internal standard. Forty m*l* of the internal standard solution was added to the sample. The exact volume of 50 m*l* from the methyl esterificated solution was filtered and placed to 500 m*l* separate funnel and extracted four times with 100 m*l* H_2O and 10 m*l* chloroform. The chloroform layer was passed through anhydrous sodium sulfate to remove moisture. The final volume of the chloroform was collected in 50 m*l* volumetric flask.

Barbaloin

To analyze barbaloin,[9] 1 g of the pulverized samples was dissolved in 100 m*l* anhydrous methanol solution and placed for eight hours at room temperature, and then filtrated. The volume of the filtrate was made to 100 m*l*, finally.

Gas chromatography[2] and high performence liquid chromatography[10,19]

Fatty and organic acids were analyzed by GC (Hewlett Packard 5890A) with FID and a 5% silar 10C 1.8× 2 mm glass column. The oven temperature was programed from 80℃ to 230℃ at the rate of 5℃/minute and then held at 230℃ for 20 minutes. The injector and detector temperatures were 220℃, and nitrogen gas was used as a carrier gas at a flow rate of 2 m*l*/minute. Injection volume was 0.5 μ*l*. The organic and nonvolatile fatty acids were identified by comparing with standard peaks and quantified with an internal standard, glutaric methyl alcohol.

Barbaloin was analyzed by Waters 510 HPLC with Waters UV detector at 254 nm and μ-Bondapac C_{18} 125 Å 10 μ column (3.9×300 mm). The mobile phase was 50% methanol and flow rate was 1.4 m*l*/min, Waters 746 Data Module was used as an integrator. Injection volume was 10 μ*l*, and retention times and peak areas of each components were compared with those of the standard solution. The barbaloin produced in Nacalai Chemicals (Japan) was used as a standard.

Results and Discussion

Seven nonvolatile fatty acids and six organic acids were separated and quantified in various species of aloe as shown in Table 2. Among them, citric, succinic and malic acid are related to raw material for cosmetic, health food and medicine. Especially, myristic acid and oleic acid make good use for cosmetic and surfactant.[7]

In fatty acids, the contents of palmitic and eicosanoic acids were comparatively abundant as shown in Table 2 and six fatty acids with the exception of linoleic acid

Table 1. Chemical and physical properties of the field soil for cultivation of aloe samples

pH (1 : 5)	O.M. (%)	T-N (%)	Ava. P_2O_5 (ppm)	Exchangeable cation (me/100 g)				CEC (mg/100 g)
				Ca	Mg	K	Na	
6.1	3.9	0.01	100.8	4.0	4.5	3.9	1.1	9.1

were distributed in five aloe species. But the contents of each fatty acid varied in the aloe species. The total content of fatty acids in *Aloe arborescence* variant 1 was highest (12.86 mg/g), and was lowest in *Alo saponaria* (6.20 mg/g).

In case of six organic acids, oxalic, malic and citric acid were distributed in most of the aloe species, and there was a great difference among the species. The content of oxalic acid was 2.83 mg/g in *Aloe saponaria*, while *Aloe arborescence* which contained 23.54 mg/g. 22. 17 mg/g of malic acid was in *Aloe arborescence* and 67. 58 mg/g in *Aloe saponaria*. The concentration of citric acid was 4.43 mg/g in *Aloe arborescence* variant 2 and 23.56 mg/g in *Aloe saponaria*. Even though the contents of other organic acids were low, they were distributed in most of the aloe species with the exception of *Aloe saponaria*. The total amount of organic acids was different depending on the aloe species, which was 51.51 mg/g in *Aloe arborescence* variant 1 and 93.97 mg/g in *Aloe saponaria*.

As shown in Table 3, the changes of fatty acids according to the hot drying conditions were little. The total contents of seven fatty acids somewhat increased to the sequence of 65℃<80℃<freeze-drying. The contents of organic acids between the samples dried at 65℃ and 80℃ were little changes, but the difference of the organic acids between freeze-dry and 65℃ air circulation, oxalic acid was 6.43, 12.33 mg/g, malic acid was 37.85, 47.25 mg/g, and citric acid was 12.87, 16.5 mg/g, respectively. The contents of organic acids in hot air circulation were higher compared to freeze-dried samples. It was suggested that organic acids were produced by the degradation of their precussors.

In the case of the contents of fatty acids in the various parts of the *Aloe vera*, the components were distributed more in the skin compared to the gel part. In the contents of total fatty acids, the gel part dried at 65℃ was 3.86 mg/g, but the skin part was 8.40 mg/g, respectively. The changes of the organic acids were similar to fatty acids, but only a large amount of malic acid was contained in the gel part. The gel part was 111.10 mg/g and the skin part was 37.84 mg/g in the case of 65℃ drying condition. And the gel part was 83.09 mg/g and the skin part was 39.09 mg/g in the sample dried at 80℃. Accordingly the contents of malic acid in the gel part was much more abundant compa-

Table 2. Contents of fatty and organic acids in the whole leaf of various aloe species (Unit : mg/g DW)

Acids	Components	Species*				
		Aloe vera	*Arborescence*	*Arborescence* variant #1	*Arborescence* variant #2	*Aloe saponaria*
Fatty acids	Myristic acid	trace	0.16	0.14	0.21	−
	Palmitic acid	0.30	2.37	3.26	1.75	1.74
	Stearic acid	0.30	0.33	0.32	0.16	0.24
	Eicosanoic acid	3.33	3.17	1.67	1.64	1.04
	Oleic acid	0.90	0.32	0.44	0.36	0.17
	Linoleic acid	trace	2.30	trace	trace	trace
	Linolenic acid	trace	0.10	7.03	4.03	3.01
	Total	6.93	8.75	12.86	8.15	6.20
Organic acids	Oxalic acid	10.59	23.54	14.32	19.07	2.83
	Malonic acid	1.06	0.41	0.79	0.59	−
	Malic acid	47.85	30.36	22.17	54.22	67.58
	Succinic acid	0.34	0.22	0.18	0.03	−
	Fumaric acid	−	0.02	0.03	0.04	−
	Citric acid	17.94	9.23	14.02	4.43	23.56
	Total	77.78	63.78	51.51	78.38	93.97

− : Not detected.
*Dried at 80℃ air circulation.

Table 3. Contents of fatty and organic acids in various parts of the *Aloe vera* dried at different temperatures (Unit : mg/g DW)

Acids	Components	65℃ air circulation			80℃ air circulation			Freeze-dry
		Whole leaf	Skin	Gel	Whole leaf	Skin	Gel	Whole leaf
Fatty acids	Myristic acid	0.09	0.11	0.01	trace	trace	–	0.08
	Palmitic acid	2.36	2.81	1.31	2.40	2.62	0.05	2.03
	Stearic acid	0.43	0.35	0.32	0.30	0.50	0.38	0.66
	Eicosanoic acid	3.25	4.21	1.78	3.33	3.81	1.04	2.27
	Oleic acid	0.96	0.92	0.44	0.90	0.77	0.29	0.65
	Linoleic acid	trace	trace	trace	trace	trace	trace	trace
	Linolenic acid	trace	trace	trace	trace	trace	trace	trace
	Total	7.09	8.40	3.86	6.93	7.70	1.76	5.69
Organic acids	Oxalic acid	12.33	12.87	2.53	10.59	11.79	1.48	6.43
	Malonic acid	1.09	1.13	0.50	1.06	1.08	–	0.61
	Malic acid	47.25	37.84	111.10	47.85	39.09	83.09	37.85
	Succinic acid	0.26	0.32	1.47	0.34	0.27	0.12	0.12
	Fumaric acid	–	–	–	–	–	–	–
	Citric acid	16.53	21.37	7.58	17.94	17.20	2.22	12.87
	Total	77.56	73.53	123.18	77.78	69.43	86.91	57.88

– : Not detected.

Table 4. Contents of barbaloin in the whole leaf of the aloe species* (Unit : mg/g DW)

Extract method	*A. vera*	*A. arborescence*	*A. arborescence* variant #/1	*A. arborescence* variant #2	*A. saponaria*
MeOH (Room temp.)	3.95	8.94	10.70	3.12	–

– : Not detected.
*Dried at 80℃ air circulation.

Table 5. Contents of barbaloin in the various parts of the *Aloe. vera* dried at different temperatures (Unit : mg/g DW)

Extract method	65℃ air circulation			80℃ air circulation			Freeze-dry
	Whole leaf	Skin	Gel	Whole leaf	Skin	Gel	Whole leaf
MeOH (Room temp.)	6.23	4.48	3.40	3.95	4.51	2.06	10.43

red to the skin part.

The contents of barbaloin among the aloe species showed great differences (Table 4). The contents of barbaloin in *Aloe vera* was 3.95 mg/g and *Aloe arborescence* variant 1 was 10.70 mg/g which was the most abundant. But barbaloin was not detected in *Aloe saponaria.*

The contents of barbaloin in the various parts of the *Aloe vera* dried at different dry conditions were shown in Table 5. The barbaloin in the skin part showed a greater concentration than that of the skin part. According to the dry conditions, barbaloin was 10.43 mg/g in the whole leaf dried by freeze-dry, 6.23 mg/g in the sample dried at 65℃, and 3.95 mg/g at 80℃. As the drying temperature was increased, the contents of barbaloin decreased.

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乾燥條件에 依한 알로에의 部位別, 品種別 脂肪酸 有機酸 및 Barbaloin 成分 硏究

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초록 : 건조조건에 의한 알로에의 품종별 및 부위별로 지방산 및 유기산과 약효성분인 barbaloin을 추출 분리정량 하였다. 7종의 지방산 및 6종의 유기산과 barbaloin 성분을 확인한 후 각 품종별, 부위별, 건조온도별로 함량을 비교하였다. 7종의 총지방산량으로 볼때 palmitic, eicosanoic acid 함량이 높았으며, *Aloe saponaria*가 가장 적었다. 6종의 유기산 총량으로는 malic, citric 및 oxalic acid가 다른 유기산보다 많았으며, *Aloe saponaria*가 함량이 많았고, *Aloe arborescence* 변종 1은 적었다. 65℃와 80℃ 열풍건조시료는 유기산 및 지방산의 함량이 거의 유사하였고, 동결건조한 시료는 열풍건조한 시료보다 함량이 낮았다. Barbaloin의 경우 품종별 함량은 *Aloe arborescence* 변종 1이 가장 높았으며, 건조조건에 따른 *Aloe vera*의 barbaloin 성분은 65℃보다 80℃의 경우 감소되었고 동결건조가 가장 많은 함량을 나타내었다.