

[CONTRIBUTION FROM THE RESEARCH LABORATORY OF McLAUGHLIN GORMLEY KING COMPANY]

STUDIES ON PYRETHRUM FLOWERS. II. THE RELATION BETWEEN MATURITY AND PYRETHRIN CONTENT

BY C. B. GNADINGER AND C. S. CORL

RECEIVED JULY 23, 1929

PUBLISHED FEBRUARY 6, 1930

Handlers of *Pyrethrum* flowers have quite generally held the opinion that the immature or closed flowers have greater insecticidal power than open or mature flowers. So firmly fixed has this idea become, that closed Dalmatian flowers command a premium of five to six cents a pound over half-closed flowers, which in turn, cost two to three cents a pound more than open flowers. At the present time, Dalmatian open flowers are slightly higher in price than Japanese flowers, which are not graded as closed and open.

In a previous paper¹ the writers described a method for determining the percentage of active principles in *Pyrethrum* flowers. By the application of this method to flowers in different stages of maturity, it has been possible to determine the comparative value of open and closed flowers.

Experimental Part

Difficulty was experienced in obtaining suitable material, because *Pyrethrum* flowers are not grown commercially in the United States. Through the courtesy of Mr. E. B. Fischer of the College of Pharmacy, University of Minnesota, a plot of *Pyrethrum roseum* was made available for this work. Samples were collected from these plants, which were three to five years old, at intervals throughout the flowering season, and the flowers were carefully shade dried. The analyses of the samples are presented in Table I.

Before the buds were formed, the roots, leaves and stems did not contain any pyrethrins. As the buds formed and developed, their pyrethrin content gradually increased. The last and most mature sample contained four times the percentage of pyrethrins found in the unexpanded buds. Samples four and six represented closed flowers at the same stage of development but collected at different times; sample six contained considerably more active material than number four, indicating that the pyrethrin content depends on the maturity of the plant as well as on the development of the flowers. The average weight of the open flowers was two to three times that of the closed flowers, and the pyrethrin content of the former was about double that of the closed flowers. Therefore, the yield of pyrethrins from a given unit of land can be approximately quadrupled by merely allowing the flowers to mature.

¹ Gnadinger and Corl, THIS JOURNAL, 51, 3054 (1929).

TABLE I
ANALYSES OF PYRETHRUM ROSEUM AT DIFFERENT STAGES OF MATURITY

Date collected	No.	Description	Av. wt., g.		Diam., mm.		Moisture in dry flowers, %	Pyrethrins (dry-basis), %
			Fresh	Dried	Fresh	Dried		
May 7	1	Roots						
May 7	2	Entire plant except roots; buds extremely small						
May 23	3	Unexpanded buds; ray florets not showing						
May 30	4	Closed flowers; ray florets visible but not expanded						
June 5	5	Half-open flowers; disk florets visible; ray florets partly expanded						
June 11	6	Closed flowers; same description as No. 4						
June 11	7	Open flowers; ray florets completely expanded; disk florets partly expanded						
June 21	8	Open flowers; ray florets expanded; disk florets expanded						
June 28	9	Open flowers; completely expanded; last of crop						
No.			Fresh	Dried	Fresh	Dried		
1			None
2			None
3			0.138	0.023	5-10	3- 7	4.6	0.22
4			.304	.052	6-13	4- 9	5.6	.40
5			.391	.086	6-14	5-11	4.9	.54
6			.285	.057	6-11	4- 9	4.9	.56
7			.502	.106	6-15	5-12	6.0	.67
8			.675	.154	9-20	7-15	6.0	.78
9			.400	.096	8-16	6-12	10.0	.87

Growing plants were not available for similar work on *Pyrethrum cinerariaefolium*, but five samples of flowers of known origin were examined in the following manner: each sample was thoroughly mixed and the

TABLE II
COMPARATIVE PYRETHRIN CONTENT OF OPEN AND CLOSED FLOWERS (*Pyrethrum cinerariaefolium*)

No.	Kind of flowers	Part of sample analyzed	Diam. of flowers, mm.	Av. wt. of flowers, g.
20	Japanese	Original sample
20	Japanese	Closed flowers	6-9	0.109
20	Japanese	Open flowers	9-15	.208
25	Japanese	Original sample
25	Japanese	Closed flowers	4-9	.101
25	Japanese	Half-closed flowers	8-12	.193
25	Japanese	Open flowers	11-14	.238
23	Dalmatian	Original sample
23	Dalmatian	Closed flowers	4-8	.064
23	Dalmatian	Open flowers	8-12	.143
24	Dalmatian	Original sample
24	Dalmatian	Closed flowers	5-8	.074
24	Dalmatian	Open flowers	10-14	.178
26	American	Original sample
26	American	Closed flowers	6-8	.086
26	American	Open flowers	9-15	.135

TABLE II (Concluded)

No.	Composition of original sample, %					Pyrethrin content, %	Ratio pyrethrin content closed:open
	Closed	Half-closed	Open	Stems	Debris		
20	9.8	20.0	64.0	0.4	5.8	0.96	
20	0.0	0.0	.80	
20	0.0	0.0	.94	1:1.18
25	36.0	40.0	16.0	1.2	6.8	.84	
25	0.0	0.0	.76	
25	0.0	0.0	.95	
25	0.0	0.0	.97	1:1.28
23	68.4	16.0	12.1	0.7	2.8	.53	
23	0.0	0.0	.61	
23	0.0	0.0	.81	1:1.32
24	53.2	24.4	16.6	2.1	3.7	.52	
24	0.0	0.0	.48	
24	0.0	0.0	.71	1:1.48
26	26.4	17.5	52.4	2.9	0.8	.85	
26	0.0	0.0	.64	
26	0.0	0.0	1.03	1:1.61

percentage of closed, half-closed and open flowers was determined. A portion of the original sample was then ground and analyzed. From the remainder of the original sample, the closed and open flowers were carefully separated, selecting only complete flowers and rejecting those from which parts were missing. The pyrethrin content of the closed and open flowers was determined. The results obtained are compared in Table II.

The open flowers contained from 18 to 61% more active principle than the closed flowers from the same lots.

It is well known that certain parts of the *Pyrethrum* flower-head contain more active principle than others,² but the distribution of the pyrethrins in the different flower parts has not been determined accurately heretofore. A sample of uncompressed Japanese flowers was carefully separated into the principal parts, using for this purpose flowers from which none of the parts were missing. The analyses of these parts are given in Table III.

TABLE III

DISTRIBUTION OF ACTIVE PRINCIPLE IN JAPANESE PYRETHRUM CINERARIAEFOLIUM

Sample No. 25	Composition of flowers, %	Pyrethrins, %	Percentage of total pyrethrins
Achenes	34.2	2.27	92.4
Receptacles	11.3	0.26	3.5
Involucral scales	11.5	0.15	2.0
Disk florets	25.8	Trace	..
Ray florets	17.2	Trace	..
Original sample	..	0.84	..
Stems	..	0.15	..

² U. S. Dept. Agr., "Insect Powder," Bulletin 824 revised, 1926.

The achenes contained 92.4% of the total pyrethrins; the receptacles and scales contained 3.5 and 2.0%, respectively, while the disk and ray florets contained mere traces. Analysis of the original sample showed 0.84% of pyrethrins, while calculation of the total pyrethrin content from the analyses of the parts gave a pyrethrin content of 0.82%. It would seem, therefore, that a high pyrethrin content depends on the development of the achenes.

The fact that the receptacles and scales and other outer parts of the flower-head contain only a small proportion of pyrethrins is of some importance. Should an attempt be made to increase the apparent pyrethrin content, obtained by the use of the method,¹ by coating or spraying flowers with a petroleum-ether soluble, copper reducing material, the adulteration could be detected by the high pyrethrin content of the outer parts of the flower. This is apparent from Table IV, in which the pyrethrin content of the outer parts of five samples of flowers is reported.

TABLE IV
PYRETHRIN CONTENT OF RECEPTACLES AND INVOLUCRAL SCALES

No.	Kind of flowers	Pyrethrins, in recept., ^a %	Recept. in flowers, %	Percentage of total pyrethrins in recept. ^a	Pyrethrins in orig. flowers, %
20	Japanese	0.27	25.0	7.0	0.96
25	Japanese	.20	22.8	5.4	.84
25	Japanese (closed)	.26	30.8	10.5	.76
23	Dalmatian	.17	31.1	10.0	.53
24	Dalmatian	.17	30.0	9.8	.52

^aScales included.

Summary

Contrary to popular belief, the pyrethrin content increases as the flowers mature, so that open flowers contain more active principle than closed flowers from the same plants. There is also some evidence that the earlier flowers produced contain less pyrethrins than the later flowers but this was not proved conclusively. Open flowers from commercial samples contained from 18 to 61% more active principle than closed flowers from the same lots.

The average weight of the open flowers is about double that of closed flowers. Therefore, the pyrethrins produced from a given tract of land can be increased three to four times by merely allowing the flowers to mature.

The roots, stems and leaves contain no pyrethrins before the flower buds are formed.

In the sample analyzed the achenes contained more than 90% of the active material. The receptacles and involucre scales contained most of the remainder. The disk and ray florets yielded only traces of pyre-

thrins. The low pyrethrin content of the outer parts of the flower is important because it affords a means for detecting certain types of adulteration.

MINNEAPOLIS, MINNESOTA

[CONTRIBUTION FROM THE RESEARCH LABORATORY OF McLAUGHLIN GORMLEY KING COMPANY]

STUDIES ON PYRETHRUM FLOWERS. III. THE PYRETHRIN CONTENT OF DIFFERENT COMMERCIAL VARIETIES

BY C. B. GNADINGER AND C. S. CORL

RECEIVED JULY 23, 1929

PUBLISHED FEBRUARY 6, 1930

The three species of *Pyrethrum* flowers that have insecticidal value are *Pyrethrum cinerariaefolium*, *Pyrethrum roseum* and *Pyrethrum carneum*; of these *Pyrethrum cinerariaefolium* is by far the most important. In 1928 more than eleven million pounds of *Pyrethrum cinerariaefolium* were imported into the United States from Europe and Japan, the latter country supplying about five times as much as all other sources combined. The amount of *Pyrethrum roseum* imported is negligible, but it is widely grown in this country for its ornamental flowers. *Pyrethrum carneum* is of no commercial importance.

The two principal commercial varieties of flowers, Dalmatian and Japanese, belong to the species *Pyrethrum cinerariaefolium*. There are several commercial grades of these flowers and it has been shown¹ that the trade preference for certain grades is unwarranted. The purpose of this paper is to show the comparative value of the different commercial varieties of *Pyrethrum* flowers.

Experimental

Twenty-eight commercial samples of whole flowers were collected from dealers in the United States and also from agents in Japan and Europe. Twenty samples of powdered commercial flowers were kindly supplied by Dr. C. C. McDonnell, Chief, Insecticide Control, United States Department of Agriculture. These powders had been kept in air-tight containers for two or three years. Two samples of *Pyrethrum cinerariaefolium* grown in Virginia were obtained through the courtesy of Dr. A. F. Sievers, Senior Biochemist, Bureau of Plant Industry, United States Department of Agriculture. Five samples of Minnesota and Iowa grown *Pyrethrum roseum* were collected and ten samples of ground or powdered commercial lots were obtained from large manufacturers of *Pyrethrum* sprays. Samples of *Pyrethrum carneum* could not be obtained.

The flowers were examined to determine the proportion of closed, half-closed, and open flowers and stems. Some of the Japanese flowers were com-

¹ Gnadinger and Corl, THIS JOURNAL, 52, 680 (1930).