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SCIENCE PAPERS AND DISCUSSIONS

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THE PREPARATION OF DRY EXTRACTS OF CASCARA

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A DRY extract of cascara sagrada has been official in the British Pharmacopœia since 1885. The first method of extraction was to macerate the bark in No. 40 powder for 48 hours with proof spirit (57 per cent. w/v of ethanol) and then to pack the moist powder into a percolator and percolate with water to a stated volume. The resulting percolate was evaporated on a water bath until a suitable consistency was obtained.

In 1898 the directions were altered to percolation to exhaustion with distilled water and evaporating to dryness on a water bath. This method was retained virtually unchanged in the 1914 Pharmacopœia. The 1932 B.P. directed that the liquid obtained by percolating the bark to exhaustion must be evaporated to dryness under reduced pressure.

In 1948 the official directions were amended to allow the percolate to be evaporated to a viscous liquid at atmospheric pressure, before completing the evaporation to dryness under reduced pressure at a temperature not exceeding 100° C. The same method is included in the 1953 Pharmacopœia.

It is generally considered that the reason for the use of reduced pressure in the preparation of this extract is to render the final product in a suitable form for the preparation of granules and not for reasons of conserving activity—hence the alteration of the method of preparation in 1948.

The method official in the United States Pharmacopeia XIV is to macerate cascara in coarse powder with boiling water for three hours followed by percolation to dryness with boiling water. The resulting percolate is evaporated to exhaustion, reduced to a fine powder and is mixed with sufficient starch to give a specified weight. This method was allowed as an alternative to the official 1932 B.P. method by the Sixth Addendum (1943) to permit the use of extracts obtained from America during the war.

Greco and Dumez[1] have published a modification of this method using a pressure cooker for the preparation of liquid extract of cascara and we have investigated their method and some modifications of it for both dry and liquid extract of cascara.

Until 1948 the only criteria for the quality of cascara extracts were their

physical properties, such as colour, taste and smell. The Pharmacopœia of that year introduced a minimum requirement of 80 per cent. of water-soluble extractive matter and this test is retained in the 1953 edition.

Fairbairn *et al.*[2,3,4,5] have now devised chemical and biological assays for several of the anthraquinone drugs and these have now been applied to cascara and its extracts[6].

As a result of assays of senna extracts Fairbairn and Michaels[7] found that the glycosides of senna are damaged by prolonged heat and Fairbairn[8] suggests that the same may be true of cascara.

We, therefore, decided to compare the activity of extracts prepared by the methods of the 1932 B.P., the 1953 B.P., Greco and Dumez's modification of the U.S.P. XIV method, and a simplification of this method devised by one of us (W. B.).

Experimental

4 extracts were prepared from the same batch of bark:—

A. By the 1932 B.P. method.

Percolation to exhaustion with distilled water and evaporation to dryness under reduced pressure. Sample A.

B. By the 1953 B.P. method.

Percolation to exhaustion with distilled water, evaporation to a syrupy extract at atmospheric pressure, followed by final evaporation to dryness under reduced pressure. Sample B.

C. The method of Greco and Dumez.

The drug in very coarse powder is placed in a suitable vessel in an autoclave and 4 times its weight of boiling water is poured over it. The mixture is allowed to macerate for 15 minutes and is then heated for 10 minutes at 15 lbs. pressure. The material is then packed into a percolator and boiling water is passed through it until it is exhausted. The percolate is evaporated to dryness at atmospheric pressure. Sample C.

D. Bruce's modification of the method of Greco and Dumez.

The drug in very coarse powder is placed in a suitable vessel in an autoclave and 4 times its weight of boiling water is poured over it. It is then immediately heated for 10 minutes at 15 lbs. pressure. The material is drained and transferred to a tincture press and the marc is pressed as strongly as possible. The expressed liquid is added to that previously drained from the drug and the mixed liquids are evaporated to dryness under atmospheric pressure. Sample D.

These extracts and the bark from which they were made were assayed chemically and, in some instances, biologically and the results are shown in Table I.

Each extract was prepared from 500 g. of bark and the yield of extracts are recorded in Table I; it is thus possible to calculate the amount of glycosides originally in the bark which has been retained in the final extracts. These proportions are given in the last column of the table.

TABLE I

CHEMICAL AND BIOLOGICAL ASSAYS OF DRY CASCARA EXTRACTS, EACH PREPARED FROM 500 G. OF CASCARA BARK

SAMPLE	YIELD OF EXTRACT	CHEMICAL ASSAY mg. of glycoside (as aloe emodin) per g.	BIOLOGICAL ASSAY mg. per g. as sennosides A and B	B/C* RATIO	AMOUNTS OF GLYCOSIDES PRESENT IN EXTRACTS	PERCENTAGE OF GLYCOSIDES EXTRACTED
Cascara bark ..	—	7·30, 7·42 } 7·36	—	—	3·68 g.	—
A B.P. 1932 method. 10 l. of percolate	100 g.	7·37	4·86	0·65	0·74 g.	20·04
B B.P. 1948 and 1953 method	122 g.	10·8, 10·8, 10·8, 10·8 } 10·8	6·60	0·611	1·32 g.	35·82
C Method of Greco and Dumez. 9 l. of percolate	143 g.	18·4, 19·0 } 18·7	9·96	0·53	2·67 g.	72·69
D Bruce's method ..	107 g.	10·8, 10·8 } 10·8	—	—	1·15 g.	31·40

* For explanation of B/C ratio *see* Fairbairn and Mahran (this Journal, p. 827).

DISCUSSION

From the results it appears that percolation with boiling water is a more efficient method of extracting cascara than with cold water. The use of autoclaving as suggested by Greco and Dumez, also appears to increase efficiency of extraction.

The method of Greco and Dumez extracted 72·69 per cent. of the glycosides present in the bark compared with 20·04 per cent. by the 1932 B.P. method and 35·82 per cent. by the present official method. Bruce's method extracted 31·4 per cent. and is, therefore, more efficient than the 1932 B.P. method. The Greco and Dumez method shows some saving of time compared with the official method. Bruce's method is very convenient and results in a very great saving of time compared with the other methods and our results show that it gives as good an extract as the official method. This extract was only assayed chemically and gave the same value as the official method with a slightly smaller yield.

The use of reduced pressure in evaporating the extracts to dryness does not result in a more potent product, and, in fact, an extract prepared by evaporation entirely under reduced pressure was slightly less potent than one where reduced pressure was only used in the final stages. This may be due to the greater time necessary to remove all the water when using reduced pressure.

Another possible reason for the greater potency of extracts prepared by the autoclaving method may be that this treatment inactivates enzymes which may cause decomposition of the glycosides. This may also be the reason for the lower potency of extracts prepared by evaporating the percolate entirely under reduced pressure. The lower temperature might

conserve the activity of such enzymes. This explanation would not, however, account for the greater yield of the autoclaving method.

Summary

1. The efficiency of 4 methods of preparing dry extract of cascara is compared.
2. The use of boiling water and autoclaving gives more efficient extraction than cold water.
3. The use of reduced pressure for evaporating the percolate does not give an extract with increased potency and may cause reduction in potency.
4. A method using autoclaving followed by pressing of the marc is as efficient as the official method and results in a great saving of time.

We wish to thank Dr. J. W. Fairbairn and Mr. G. E. D. Mahran for carrying out the chemical and biological assays on these extracts.

References

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