

Technology Section

Pakistan J. Sci. Ind. Res., Vol. 31, No. 12, December 1988

A MODIFIED SOXHLET EXTRACTOR FOR USE IN TOXICOLOGICAL ANALYSIS

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(Received January 6, 1988; revised May 22, 1988)

A soxhlet extractor which can be used at room temperature and under reduced pressure has been described. It is useful in toxicological analysis and for the extraction of plant materials.

Key words: Soxhlet, Continuous,, Reduced pressure.

INTRODUCTION

The continuous soxhlet extractor has long been in use. However, no attempt to use the extractor under reduced pressure has been recorded. Curry and Phang [1] devised a continuous tissue extractor. This is used at ordinary temperature and under reduced pressure, and a large quantity of tissue can be efficiently extracted. In laboratories where this extractor is not available an alternative can be found in the modified continuous soxhlet extractor described below.

The ordinary soxhlet extractor does not function under reduced pressure. Under such a condition it has been found that the soxhlet will not siphon. This difficulty is however overcome by a little modification to the ordinary soxhlet extractor.

MATERIALS AND METHODS

The glycosides were purchased from BDH Chemical Ltd., Dorset England and Sigma Chemical Company, U.S.A.

The modification to the ordinary soxhlet extractor consists of a polythene tubing connection on the lower end of the ordinary soxhlet. The lower end of this tubing dips into the solvent in the evaporation flask (Fig. 1).

Application of the modified soxhlet extractor in extracting glycosides. 2 mg of digitoxin was added to 25 gm of liver and then homogenised in alcohol to obtain a reasonably thick slurry. This was then mixed with 1 gm anhydrous sodium sulphate, put in a thimble, covered with cotton wool and introduced into the soxhlet extractor. This was then connected to a vacuum pump. Alcohol was poured into the three necked evaporation flask which was placed in a water bath maintained at 50°. A thermometer was attached to one neck of the evaporation flask while an air inlet was connected to the third. The extraction was carried out for eight hours under reduced pressure 13.3 pa (0.1 mm Hg). The alcoholic extract was evaporated to dryness in vacuo and the residue taken up in 20 ml warm

absolute alcohol. Any precipitate formed was filtered off and the filtrate again evaporated to dryness in vacuo. The residue was taken up in 10 ml 0.001 N H₂SO₄ shaken vigorously for 15 min and filtered. The filtrate was extracted with 20 ml light petroleum (60°-80°) followed by 20 ml solvent ether. Both organic phases were rejected. The aqueous phase was adjusted to pH 6 with dilute ammonium hydroxide. It was then extracted with 20 ml chloroform. The chloroform layer was removed, concentrated, purified by paper chromatography eluted and the concentration of digitoxin in the eluate determined by the method of Jelliffe [2].

Curry-Phang continuous extractor. 2 mg of digitoxin was added to 25 gm of liver homogenised and extracted for 8 hours using the Curry-Phang extractor. The extract so obtained was treated as described for the modified soxhlet extractor.

Both methods of extraction were used for the extraction of the following glycosides namely; aesculin, amyg-

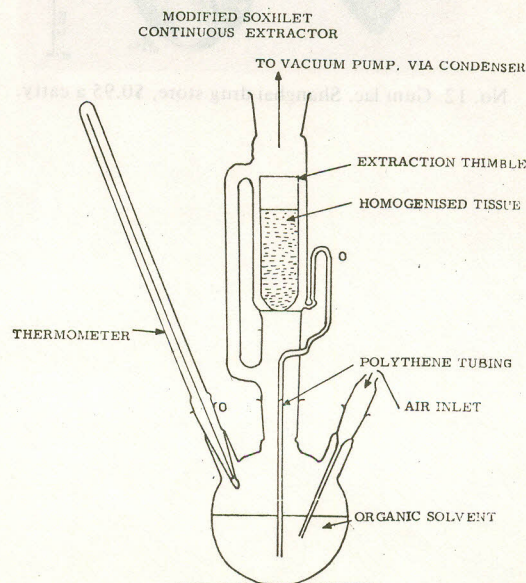


Fig. 1. Modified Soxhlet continuous extract.

dalín, digitoxin, senoside B, thevetin etc. from homogenised liver. The percentage recovery by each method was determined.

RESULTS AND DISCUSSION

The percentage recovery of each glycoside is as shown in Table 1.

From the result it can be seen that the modified soxhlet extractor gave comparative results as the Curry-Phang extractor. There are many advantages to the use of the modified soxhlet apparatus. The extraction is at room temperature and under reduced pressure. These conditions are so mild that glycosides and labile compounds are not destroyed. It has the advantage of being simple and easy to handle. In addition, it requires comparatively smaller quantity of organic solvent. It can also be used for the extraction of plant materials.

The methods were applied to the recovery of glycosides from liver homogenate. When each of the method was applied to the extraction of these different glycosides individually added to liver, the recovery was found to differ from glycoside to glycoside. The percentage recovery was found to vary from as high as 72 % for singrin through 60 % for digitoxin to as low as 20 % for aesculin, digoxin, senoside B and anthraquinone. With those glycosides which gave a low recovery, with both methods it was found that the same low results were obtained using the direct extraction method.

The low recoveries were therefore not due to any inefficiency on the part of the Curry-Phang extractor of the

Table 1.

Glycoside	Curry-Phang recovery (%)	Modified soxhlet extractor recovery (%)
Digitoxin	60	50
Thevetin	55	50
Digoxin	20	20
Amygdalin	72	70
Singrin	72	71
Solanin	40	38
Anthraquinone	20	20
Aesculin	20	20
Phloridzin	40	41
Senoside B	20	18

The values represent an average of three determinations for each of the glycosides.

modified soxhlet extractor. The 60 % recovery found for digitoxin compares well with the value obtained by Curry [1]. In addition it requires comparatively smaller quantity of organic solvent and there was no loss of alcohol by air under vacuum.

REFERENCES

1. A.S. Curry and S.E. Phang, *J. Pharm. Pharmacol.*, **12**, 437 (1960).
2. R.W. Jelliffe, *J. Lab. Clin. Med.*, **67**, 694 (1966).

MATERIAL AND METHODS

Wheat and rice straw were purchased from the local market to carry out these studies. These materials were subjected to different physical treatments as described below.

(1) Fine grinding: Wheat and rice straw were ground in a ball mill and passed through 80, 100, 120 and 200 mesh sieves.

(2) Steam/pressure treatment: The materials having 20 # moisture were autoclaved at different pressure (15-20 lb per sq inch) for different intervals of time. After the reaction time, pressure was gradually released to atmospheric pressure and then the substrate was dried at 100° for 24 hours.

(3) Dry heat treatment: The materials were evenly spread in 1/4 inch layer in enamelled trays and exposed to various temperatures in a forced draft air oven for different