Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Armstrong, B. F. and Neas, E. D.(1990) 'Development of a Microwave Distillation System for the Analytical Laboratory', Separation Science and Technology, 25: 13, 2007 — 2015
To link to this Article: DOI: 10.1080/01496399008050440
URL: http://dx.doi.org/10.1080/01496399008050440

SEPARATION SCIENCE AND TECHNOLOGY, 25(13-15), pp. 2007-2016 (1990)

DEVELOPMENT OF A MICROWAVE DISTILLATION SYSTEM FOR THE ANALYTICAL LABORATORY

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ABSTRACT

A microwave distiller was developed for use in the analytical laboratory. The microwave distiller consists of a microwave system which has approximately 1400 Watts of input power and a distillation apparatus mounted above the microwave cavity. The microwave distiller was able to reduce the distillation time required for the distillation of citrus oil and Total Kjeldahl Nitrogen by 20 and 9 fold, respectively.

INTRODUCTION

Separation by distillation has been around since at least 100 A.D. when it was used to separate alcohol for the production of brandy. Since that time there have been numerous applications for the separation of volatile liquids using distillation. These include well known applications such as separation of crude oil and discreet separations of flavors and oils from food and other samples. In many applications the time required for separation can be significant. These include such separations as ammonia for nitrogen determination, Scott oil and flavor determinations in citrus juices, diacetyl determination in fermentation processes, and phenol and cyanide determination in environmental samples.

Historically, samples are heated for distillation by conduction using an open flame, heating mantel, hot plate, or a sand, steam or oil bath. Distillation times are affected by limitations inherent within these conductive heating methods. Typically, vessels used for distillation are poor conductors of heat, therefore, time is required to first heat the vessel and then transfer that heat into the solution. Also, because of vaporization at the surface of the liquid a thermal gradient is established by convection currents and only a small portion of the sample fluid is at the temperature of the applied heat (Figure 1). Some distillations, e.g. distillation for ammonia recovery, use steam injection to decrease the distillation times. Steam distillation will reduce heating mantel distillation times from 45-60 minutes to 5-8 minutes. However, applications for steam distillation are limited to water soluble distillations where steam injection would not contaminate the sample.

Microwave heating results from direct absorption of the input energy by the sample solution. Therefore, all of the sample is heated simultaneously and surface vaporization has little effect on the temperature of the solution. Also, because the rate of heating is so much more rapid, substantial localized superheating occurs and thus part of the sample solution is slightly above the boiling point (Figure 2). Both of these advantages lead to significantly shorter distillation times. This paper illustrates a distiller that uses microwaves to heat the sample solution and includes data for the distill ation of a flavor from orange juice and ammonia from an acid dissolution.

EXPERIMENTAL

Total Kjeldahl Nitrogen Determination

Acid blanks were prepared by heating a mixure of sulfuric acid, $CuSO_4$ and K_2SO_4 to 360 C. The acid blanks were cooled and approximately 0.1 gm of ammonia sulfate was added to the solutions. Each of these ammonia sulfate standards were neutralized with hydroxide and distilled in either a steam injection distiller or the microwave distiller. The ammonia was

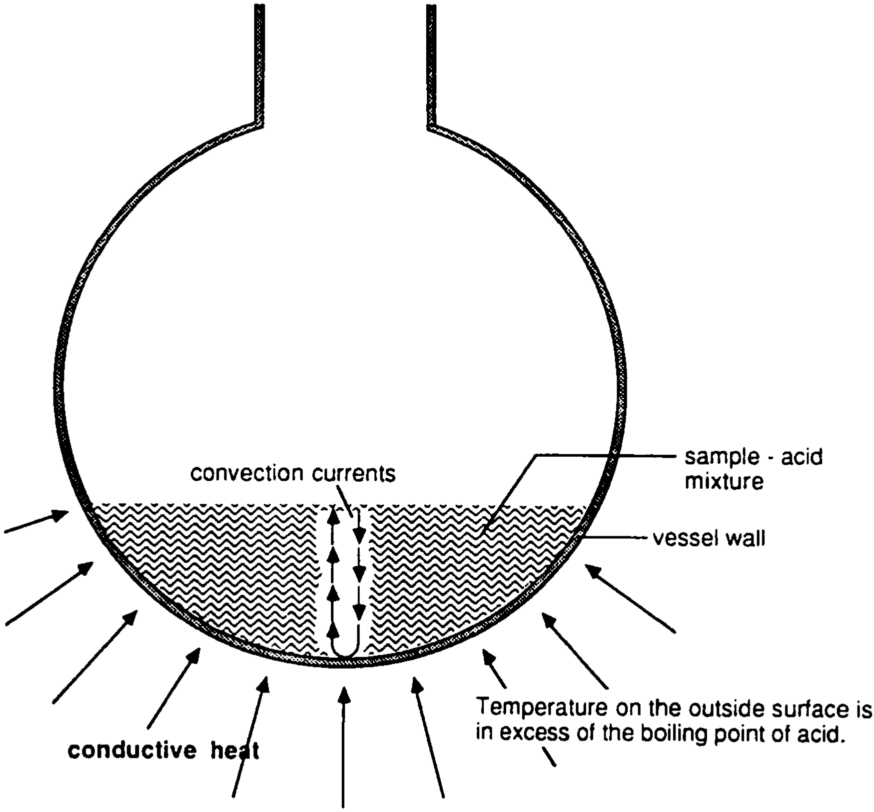

Figure 1. Schematic of sample heating by conduction

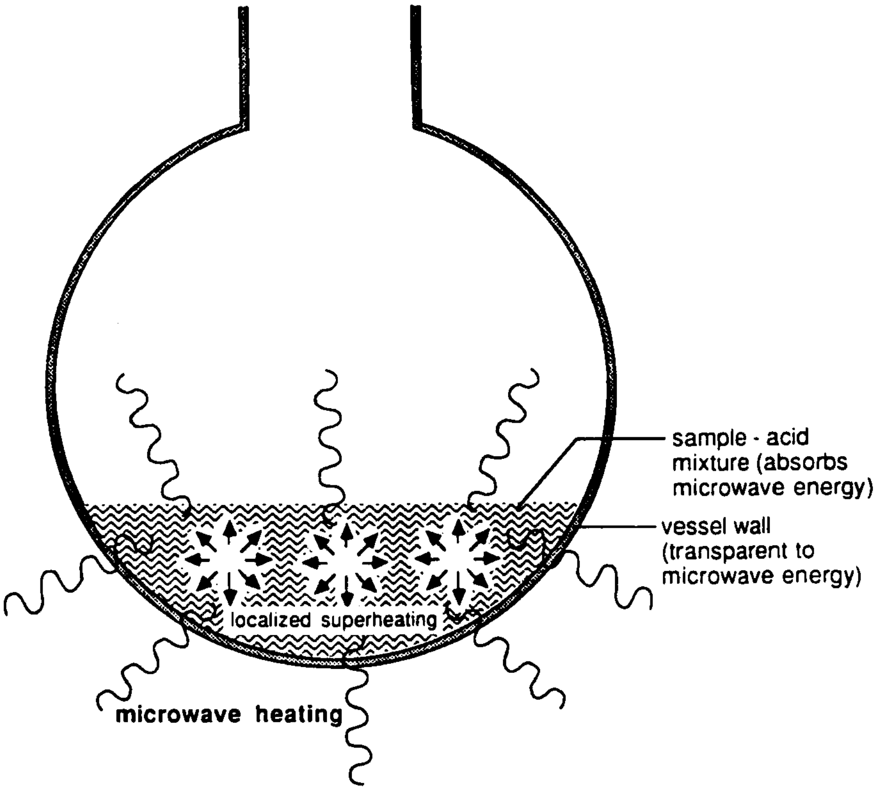

Figure 2. Schematic of sample heating by microwaves.

collected and titrated as outlined in reference 2. The percent nitrogen recovery was calculated and divided by the theoretical recovery and reported as percent recovery.

Limonene (Citrus Oil) Determination

Standard method.

Twenty-five mls of an orange juice sample was pipetted into a 1000 ml distillation flask. Afterwards, 25 ml of 2-propanol and 50 ml of water were added to the flask. The solution was heated with a heating mantel at full heat. Thirty ml of distillate were collected in a 150 ml beaker and the limonene (oil) content was determined as outlined in reference 1. Three different orange juice samples were evaluated three different times for oil content and the average and standard deviation calculated for each juice sample.

Microwave method.

The sample solution was prepared exactly as described in the standard method above and placed in the microwave distiller. The use of the microwave distiller encompasses adding the sample to a 1000 ml Pyrex boiling flask equipped with a ground glass 24/40 connector at the neck. The flask is placed into the microwave cavity and supported by a spring adjustable pedestal which allows easy entry and removal of the flask. The pedestal is placed into and taken out of the cavity along with the flask. The flask is joined to the condensing apparatus by a rigidly mounted Pyrex tube that passes out of the microwave cavity through a hole in the ceiling and connects to the boiling flask via the 24/40 connector. The distillation apparatus of the microwave distiller is illustrated in Figure 3. Thirty mls of distillate were collected in a 150 ml beaker and analyzed as described above in the standard method section.

RESULTS

Table I shows the comparative recoveries between the standard method and the microwave method for Limonene in orange juice. The recoveries were statistically equal and the distillation times were 45 seconds for microwave distillation versus 15 min for the conventional distillation method.

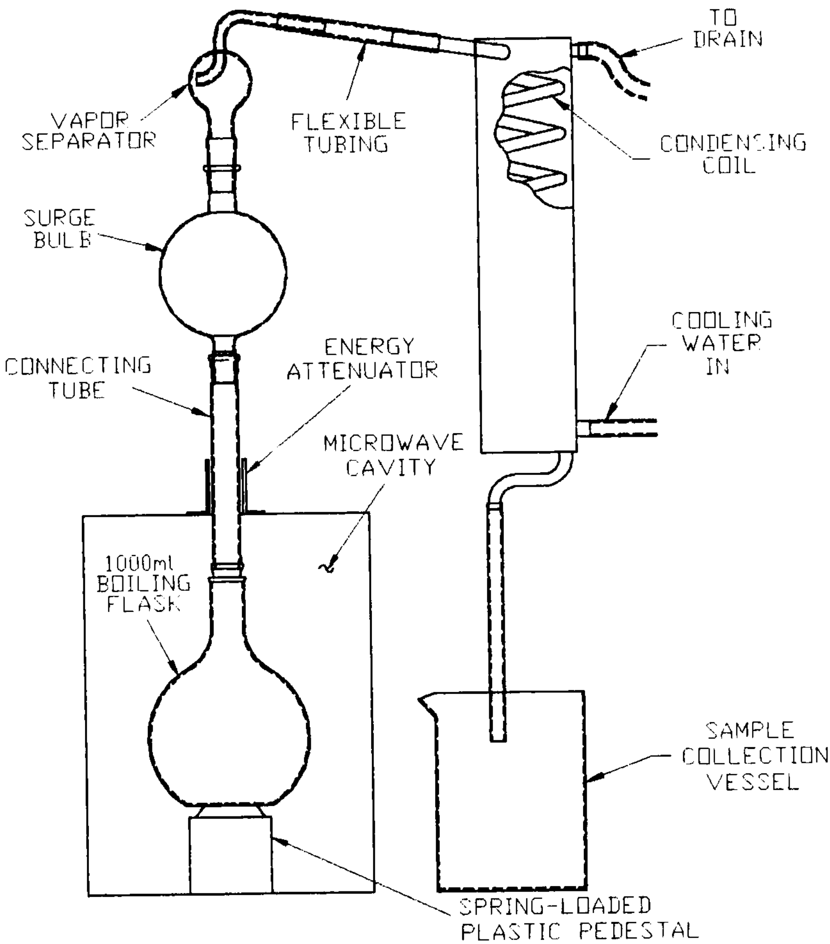

FIGURE 3. SCHEMATIC OF THE DISTILLATION APPARATUS USED IN THE MICROWAVE DISTILLER.

TABLE I. Determination of citrus oil in orange juice by the standard method and the microwave method

	Standard Method		Microwave Method	
Sample Number	Time (min)	Percent oil	Time (min)	Percent oil
1	15	0.020	0.75	0.020
2	15	0.019	0.75	0.020
3	15	0.021	0.75	0.021

Table II gives the recoveries in percent nitrogen that were obtained using a steam distiller and the microwave distiller. The times and recoveries were equal for both distillation methods.

DISCUSSION

The use of microwave heating to speed up sample preparation and analysis is now widely accepted for several applications. These include moisture analysis (3), acid digestion for atomic absorption and emission analysis (4), extraction (5), chemical synthesis (6-7), and protein hydrolysis (8-9). The microwave distiller has been used to reduce the time required for the distillation of citrus oil by 20-fold. This will allow processors to accomplish on-line determination of the oil and make adjustments while packaging. Also, microwave distillation is 9-fold faster than conventional heating methods for Kjeldahl distillations and as fast as steam injection heating. Therefore, microwave distillation is as fast as steam injection for use in aqueous distillations but does not risk contamination which might be introduced with the steam. Preliminary work (not shown) in our laboratory has indicated that microwave distillation can be used for the separation of essence from citrus juices. Also, it

TABLE 2. Determination of percent nitrogen recovery by steam distillation and microwave distillation

Sample Number	Steam Distillation Time (min)	Steam Distillation Percent Recovery	Microwave Distillation Time (min)	Microwave Distillation Percent Recovery
1	6	99.5	6	99.4
2	6	100.3	6	99.6
3	6	99.7	6	100.1

appears that microwave distillation can be used to determine the chemical oxygen demand of citrus juices. The time reduction using microwave distillation for these techniques is approximately 60 min. In summary, we feel that microwave distillation is capable of significantly reducing the time required by many of the conventional distillations used in analytical laboratories.

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