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Gypsum as a Good Stationary Phase for Separation of Some Organic Compounds in Chromatography

Amoozadeh A.
Department of Chemistry, Faculty of Science, Semnan University, Semnan, Iran.
e-mail: aliamoozadeh@yahoo.com

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Abstract

This paper describes the preparation of gypsum tlcs and shows the utility of this type of tlcs for a variety of organic compounds and finally obtains the relation between gypsum tlc and column chromatography (filled by gypsum).

Keywords: Gypsum, Thin-layer chromatography, Stationary phase, Column chromatography.

1. Introduction

Toward the end of the 1950s, thin-layer chromatography (tlc) practically replaced paper chromatography as one of the most popular chromatographic techniques. The evaluation of tlc is an excellent illustration of how new scientific improvements directly follow from the achievements of the previous contributors (Sârbu *et al.* 2001).

Today in a modern organic chemistry laboratory, tlc is used as a rapid, inexpensive and facile method for: a) determination of the number of products in a mixture or following a new reaction and/or b) obtaining the necessary data for column chromatography (Ley *et al.* 2002).

Usually, organic chemists use silica gel tlc and silica gel powder as solid phase in column chromatography that are easily available (Still *et al.* 1978, Cserháti *et al.* 2002).

In this work, at first we prepared tlc sheets by gypsum and found the different R_fs for some organic compounds with Petroleum ether: Hexane (85:15) as eluant and then, we used gypsum (Amoozadeh *et al.* 2003) as a stationary phase for separation of 4-methoxyphenol and acetanilide in column chromatography with the same eluant (Amoozadeh & Fazli 2004).

2. Experimental

2.1. Reagents

2.1.1. Gypsum: The industrial gypsum was supplied by Sepidar gypsum co. by grade 1 and 400 mesh size.

2.1.2. Organic compounds: All organic compounds were analytical grade from Sigma-Aldrich Company without further purification. Products of entries 5, 6 and 12 in Table 2 were made in our research laboratory and purified by flash chromatography.

2.1.3. Solvents: All solvents were industrial grade from Kian-Kaveh co.

2.2. General Procedure

2.2.1. Preparation of TLC Sheets: A suspension of 10 g of gypsum (400 mesh) in 20 mL of distilled water mechanically stirred for one minute. The glass support (3×8 cm^2) are gently rinsed in a horizontal position and pulled out. After drying, the tlc sheets got ready.

Table 1. The obtained column chromatography details on the gypsum

Column Diameter	Vol. of Eluant	Sample	Typical fraction size
mm	ml	mg	ml
10	100	120	5
20	200	480	10
30	400	1080	20
40	600	1920	30
50	1000	3000	50

2.2.2. Column Chromatography: At first an appropriate solvent is found which can separate the mixture and moves desired components on analytical tlc to an R_f of 0.38 and near to 1.0 respectively. Having chosen the solvent, a column of the appropriate diameter (see Table 1) is selected and a small plug of glass wool is placed in the tube connecting the stopcock to the column body. Next a smooth 3 mm layer of 50-100 mesh sand is added to cover the bottom of the column and a suspension of 400 mesh gypsum in the appropriate solvent is poured into the column in a single portion. With the stopcock open, the column is gently tapped vertically on the bench to pack the gel. The dept of the gel must be 15 cm. Excess eluant is forced out of

the column above the adsorbent bed. The top of the gypsum should not be allowed to run dry. Next a 3 mm layer of sand is carefully placed on the flat top of the gel bed. Next the sample is applied by pipette as a 20-25% solution in the eluant to the top of the adsorbent bed. By opening the stopcock, the sample is pushed into the gypsum. The solvent used to pack the column, is ordinarily reused to elute the column. The wall of the column is washed down with few milliliters of fresh eluant. The washing is pushed into the gel as before. The column is carefully filled with eluant so as not to disturb the adsorbent bed. Fractions are collected until all the solvents have been used (see table 1 to estimate the amount of solvent and fraction size). It is best not to let the column run dry since further elution is occasionally necessary. Purified components are identified by tlc. If the forgoing instructions are followed exactly, there is little opportunity for the separation to fail.

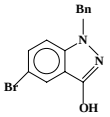
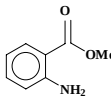
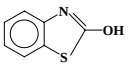
3. Results and Discussion

In the case of silica gel, for a fine separation of two compounds on column chromatography, the first R_f must be 0.33 and the second one must be near to 1($\Delta R_f \geq 0.1$) (Still & Mitra 1978). In the case of gypsum, our results show that these data should be 0.38 (Table 2 entry 3) and near to 1.0 (Table 2 entry 1) respectively. So we can separate the similar compounds on the solid phase gypsum as well as silica gel.

In addition, for the first time, we obtained the different R_fs for some organic compounds on gypsum tlcs with the same eluant. Our results show that this type of tlc could be used for a wide variety of organic compounds as they are shown in Table 2.

Because of lower cost and easier availability of gypsum and also the role of gypsum as a solid phase in chemical reactions (Kumar 1971, Wierzchowski *et al.* 1971) it is possible to use the gypsum solid phase in chromatography techniques. In comparison with silica gel and also reliable results of gypsum, this replacement is satisfactory.

Table 2. Different R_fs for some organic compounds on the gypsum tlc with Petroleum ether: Hexane (85:15) as eluant

Entry	Organic Compounds	R_f
1	4-Methoxyphenol	0.60
2	4-Aminoacetophenoxy	0.25
3	Acetanilide	0.38
4	*p*-Anisidine	0.60
5		0.15
6		0.94
7	2-Aminobenzaldehyde	0.80
8	2-Amino-5-methylbenzoicacid	0.10
9	*o*-Benzoicsulfimide	0.11
10	Oxindole	0.23
11	3,5-Diaminobenzoicacid	0.02
12		0.80
13	Hexamethylendiamine	0.20
14	4-Methyl-3-nitroaniline	0.67
15	*p*-Toluenesulfonhydrazide	0.77
16	4-(Dimethylamino)pyridine	0.12
17	1,4-Dimethoxybenzen	0.86
18	4-Nitrobenzoicacid	0.90

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