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Use of Gypsum as a Good Stationary Phase in Thin Layer Chromatography and Relation between R_fs and Dipole Moments of Some Organic Compounds

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In this paper, the preparation of gypsum TLCs and its utility for a variety of organic compounds have been reported. In addition, dipole moments of some organic compounds have been calculated by AM1 semi-empirical method and drew the R_fs of these compounds in gypsum TLCs *versus* their dipole moments. The results showed that a linear correlation exist between R_fs and dipole moments. Finally, by theoretical calculations in AM1 method, the dipole moments of some other organic compounds are obtained and their R_fs on gypsum TLCs are predicted. The experimental results on gypsum TLCs show good agreements with the theoretical results.

Key Words: Gypsum, Thin layer chromatography, Dipole moments, Organic compounds.

INTRODUCTION

Thin-layer chromatography (TLC) is a commonly used technique in synthetic chemistry for identifying compounds, determining their purity and following the progress of a reaction[1-3]. It also permits the optimization of the solvent system for a given separation problem. In comparison with column chromatography, it only requires small quantities of the compound (~ng) and is much faster as well. In thin layer chromatography (TLC), as an example of the conventional technique, the driving force for the solvent migration is the decrease in the free energy of the liquid as it enters the porous structure of the layer and the transport mechanism is the action of capillary forces. TLC is also used to support the identity of a compound in a mixture when the R_f of a compound is compared with the R_f of a known compound (preferably both run on the same TLC plate).

Toward the end of 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[4].

Today in a modern organic chemistry laboratory, TLC is used as a rapid, inexpensive and facile method for detection and determination of the number of products in a mixture or following a new reaction and obtaining the necessary data for column chromatography[5-9]. Usually, organic chemists use silica gel TLCs and silica gel powder as solid phase in column chromatography which is easily available[10].

In present study, a simple new TLC method with gypsum sheets is proposed. At first, TLC sheets using gypsum were prepared and the R_fs for some organic compounds with petroleum ether:hexane (85:15) as eluent were determined. Finally, dipole moment of the organic compounds were calculated by AM1 semi-empirical method. A graph is plotted between the R_fs of these compounds in gypsum TLCs and their dipole moments. The results showed that a linear correlation exist between R_fs and dipole moments.

In addition by the same theoretical calculations, the dipole moment of some other organic compounds were obtained and also predicted their R_fs on gypsum TLCs. The experimental results on gypsum TLCs showed a good agreements with theoretical results.

EXPERIMENTAL

The gypsum was supplied by Sepidar Gypsum Company by grade 1 and 400 mesh size. All organic compounds were analytical grade from Sigma-Aldrich Company and used without further purification.

Preparation of sulfomangenate solution: Dissolve 5 g potassium permanganate in 300 mL of distilled water and add gently 10 mL conc. sulfuric acid.

Preparation of stationary phases: Adsorbents, as employed in the present study were gypsum with 400 mesh size. Using glass plates as the inert solid support, the gypsum was layered as a matrix. 10 g of gypsum were added to in 20 mL of distilled water and stirred mechanically for 1 min. The glass support (3×12 cm^2) are gently rinsed in a horizontal position and pulled out. After drying, the TLC sheets got ready.

Thin layer chromatography: Test solutions (1.0 µL) were applied by means of micropipette about 2 cm above the lower edge of the plates. The spot was allowed to dry and then the plates were developed in the chromatographic chamber presaturated for 5 min with petroleum ether:hexane (85:15) as solvent using ascending technique. The solvent ascent was kept up to 9 cm from the point of application. After development, the plates were dried at 60 ºC followed by appropriate method for development.

Methods of development: Two methods were used for developing the spots. All of the organic compounds developed by at least one of these two methods (Table-1).

Iodine chamber development method: By putting the TLC sheets in an appropriate size of iodine chamber after about 1 min spots appeared.

Sulfomanganate development method: By plunging of TLC sheets in sulfomangenate solution for about 30 s and heating over a heater, spots appeared.

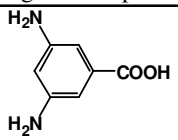
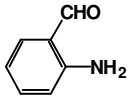
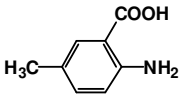
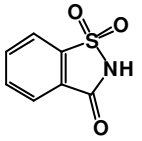
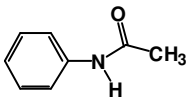
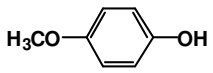
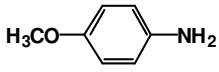
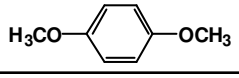
The R_f values were calculated as

R_f = Distance of trailing front / distance of leading front

RESULTS AND DISCUSSION

The R_fs values of some different compounds in a variety of organic compounds on gypsum TLCs were obtained. The spots were developing either by iodine or by sulfomanganate method (Table-1). The dipole moment

TABLE-1
DIFFERENT R_fs OF SOME ORGANIC COMPOUNDS ON GYPSUM TLCs AND THEIR CALCULATED DIPOLE MOMENTS

Entry	Organic compound	AA	BB	CC	DD
1		Good	Good	5.634	0.02
2		Good	Good	3.987	0.08
3		Bad	Good	3.069	0.19
4		Bad	Good	4.120	0.11
5		Bad	Good	2.974	0.38
6		Good	Moderate	2.404	0.54
7		Good	Good	1.893	0.60
8		Bad	Good	0.001	0.86

AA = Development by iodine method; BB = Development by sulfomanganate method; CC = Calculated dipole moment (Debye); DD = R_f on Gypsum TLC (petroleum etther/hexane, 85/15)

values of these organic compounds were also calculated using Hyper Chem. program in AM1 method (Table-1). Thus, with the help of the obtained data, a graph is plotted between calculated dipole moments and R_fs value (Fig. 1), which resulted a good linear correlation between dipole moments and R_fs on gypsum TLCs.

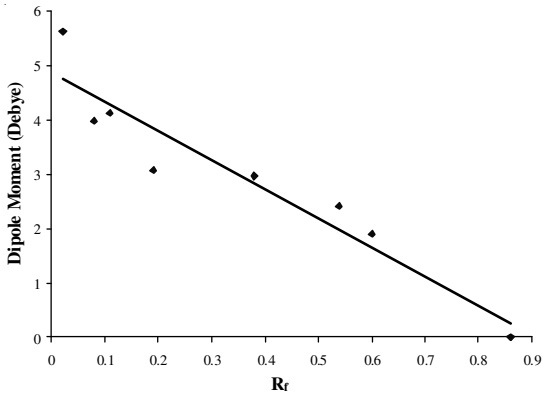
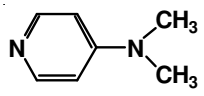
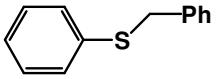


Fig. 1. Relation between calculated dipole moments of some organic compounds and their R_fs on gypsum TLCs

It is concluded that if the calculated dipole moment of any compound is known, then its R_f on gypsum TLCs can be predicted. In order to approve this hypothesis, the dipole moments of organic compounds **8** and **9** were calculated by the same calculations. The dipole moments of compounds **8** and **9** were found to be 3.708 and 1.167, respectively.



8



9

Structure of studied compounds

By using corresponding Fig. 1, the R_fs of mentioned compounds must be 0.18 and 0.693, respectively. The experimental gypsum TLCs showed that the R_fs of compounds **8** and **9** were 0.17 and 0.65.

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