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Research Article

Comparative Study for Selection of the Optimum Mobile Phase for Separation of Caffeine and Paracetamol by Using TLC Chromatographic Method

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ABSTRACT
Objective. To investigate the best mobile phase for separation of Paracetamol (PAR) and Caffeine (CAF) using TLC method. Methods. Different mobile phases which were mentioned in literature review were tried and retention times for both PAR and CAF were recorded for each experiment separately. Results : It was found that retardation factors for solvent A R_f (PAR) =0,59 and R_f(CAF)=0,90 ; for solvent B R_f(PAR)=0,92 and R_f(CAF)=0,81; R_f(PAR)=0,22 and R_f (CAF)=0,16. Solvent A is composed of n-hexane-ethyl acetate-ethanol (2.5 + 1.5 + 0.4, v/v/v), solvent B is composed of chloroform,ethyl acetate and ammonia (15,0 + 4,3 + 0,3 v/v/v) and solvent C is composed of methanol,glacial acetic acid and water (25,0 +4,3+70,7 v/v/v). Conclusion: It was found that the best solvent for separation of CAF and PAR in TLC chromatographic method was composed of n-hexane-ethyl acetate-ethanol (2.5 + 1.5 + 0.4, v/v/v).

Keywords: Paracetamol, caffeine, Mobile phase and TLC.

INTRODUCTION

Chromatography is the most used techniques to separate drug mixtures in quality control in manufacturing companies. The mixture is dissolved in a liquid called mobile phase, which carries through the structure hold another substance called stationary phase. This separation is based on the division of the difference between mobile and fixed phases[1].

Thin-layer chromatography (TLC) Chromatography is a technique used to separate mixtures of non-volatile[1]. The implementation of thin layer chromatography on a sheet of glass, plastic, or aluminum foil that coated with a thin layer of adsorbent material, usually silica gel, aluminum oxide (alumina), or cellulose.

After the sample was applied on a plate, and directs the solvent or solvent mixture (known as the mobile phase) so that the work of the panel via capillary. Because different analyzes stepping plate TLC at different rates, and achieved separation[2]. The mobile phase have different characteristics from the stationary phase. For example, with silica gel, which is very polar material, used mobile phases, such as non-polar as heptane. The mobile phase may be a mixture, allowing chemists to refine the characteristics of the bulk of the mobile phase. After the experiment, and the perception of spots. Often this can be done simply by dropping the UV light (usually at 254 nm) on paper. It can also be the chemical processes used to visualize the spots. Anisaldehyde, for example, colorful forms adducts with many vehicles, and will most sulfuric acid char organic compounds, leaving a dark spot on the paper[2].

To measure results, the division of the distance traveled by the material studied by the total distance traveled by the mobile phase. (You should not allow the mobile phase to get to the end of the fixed period.) This ratio is called the retention or RF factor. In general, a substance similar to the structure of the stationary phase with low retention factor, while it has a structure similar to the mobile phase will be high retention factor. Retention factors are characteristic, but depending on the status of the exact stage of mobile and fixed will change. For this reason, changing the composition of the mobile phase is greatly necessary to achieve acceptable separation of drugs mixture[3].

Thin layer chromatography can be used to monitor the progress of the reaction, and identify compounds in a given mixture, and determine the purity of the material[3].

The improvements on the original way to automate many steps to optimize TLC and allow for quantitative analysis more precise accuracy is referred to as the HPTLC method, or "high-performance TLC"[3].

Caffeine(CAF) (Fig1) is considered neural stimulant drug of the methylxanthine class[4]. Usually people drink worldwide with psychoactive stimulation effect[5]. Paracetamol (PAR), also known as acetaminophen[4] is a medicine used to treat pain and high temperature. It is usually used for mild to moderate pain[5].

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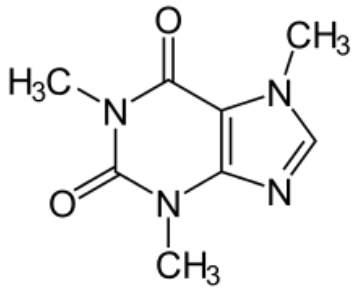

Figure 1: showing structure of caffeine.

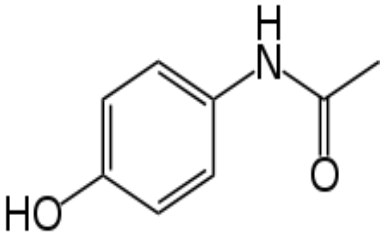

Figure 2: showing structure of paracetamol.

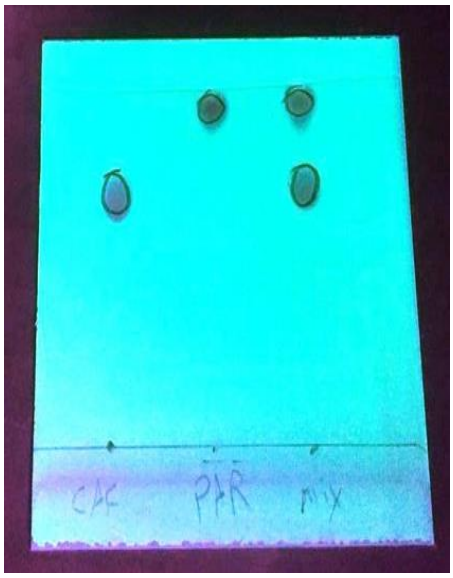

Figure 3: showing separation of paracetamol and caffeine by system A.

Figure 4: showing separation of paracetamol and caffeine by system B.

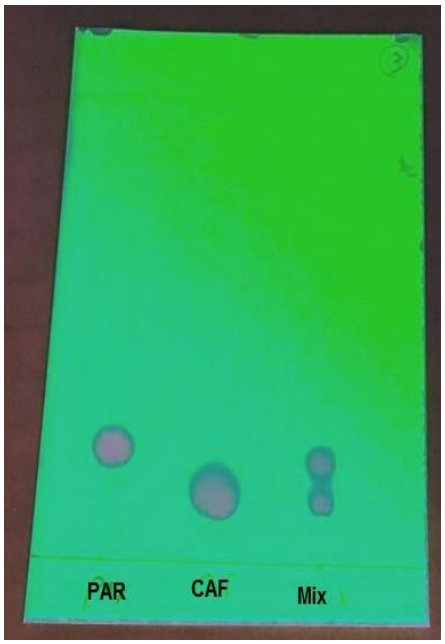

Figure 5: showing separation of paracetamol and caffeine by system C.

However, combination of both drugs are very common worldwide especially in common cold treatment. Many methods had been mentioned in literature review for analysis of mixture of CAF and PAR.

A simple and efficient method[6] using TLC with a fluorescence plate reader has been described by Tavallali etal in 2010for simultaneous determination of CAF and PAR. Determination was carried out using the fluorescence-quenching action of CAF and PAR on a TLC plate with a fluorescent indicator at lambda ex = 254 nm in the linear ranges of 0.2-1.9 and 0.03-1.5 µg/L, respectively. Separation of CAF and PAR were performed on the TLC plate, and the best results were obtained using the optimized mobile phase n-hexane-ethyl acetate-ethanol (2.5 + 1.5 + 0.4, v/v/v).

The second method for Separation of CAF and PAR were performed on the TLC plate by Caitlin Sullivan and Joseph Sherma in 2003, and the results were obtained using the mobile phase chloroform,ethyl acetate and ammonia (15 + 4:3 + 0,3 v/v/v)[7]

The third method for Separation of CAF and PAR were developed by Soponar et al in 2009 on the TLC plate, and the results were obtained using the mobile phase methanol, glacial acetic acid and water (25 +4,3+70,7 v/v/v)[8]

The aim of the experiment is to select the best resolving system (mobile phase) for separation of CAF and PAR.

Experiments

Apparatus, glass jar, UV lamp (254 nm) in cabinet, TLC plate (Merck, KGaA, Dernastate, Germany)

Material and reagents (caffeine, paracetamol powder SDFCL, fine-chem limited, Mumbai-30, India)
All reagents and chemicals used were of analytical grade and were used without any purification [n-hexane, ethyl acetate, ethanol, chloroform, ammonia, methanol, glacial acetic acid and water]. All were purchased from company lobal chemie, New Delhi, India.
Preparation of standard solutions
Nearly 0.1 gram of each drug was weighted and transferred into a two separate 100-mL volumetric flask then 100 mL methanol was added and shacked till dissolve in sonicator .
Procedures

- Standard solutions were prepared as mentioned above and mobile phases were prepared as shown in introduction
- A line with pencil was drawn at the base of the plate 1 cm apart from the edge
- the two drugs were applied on TLC plate by micro pipette
- Drying of spots: the plate was left in atmosphere at room temperature till drying of spots
- The mobile phase was left for 20 minutes in the jar for saturation of air.
- Devlopemnt of experiment was done by running the selected mobile phase on TLC plate just before the other edge by 1 cm
- Drying of developed plate: the plate was left in atmosphere at room temperature till drying.
- The developed TLC plate was examined under UV lamp (254 nm)

RESULT AND DISCUSSION

The following results were recorded for each mobile phase:
A-R_f (PAR) =0,59 R_f(CAF)=0,90 See fig:3
B- R_f(PAR)=0,92 R_f(CAF)=0,81 See fig:4
C- R_f(PAR)=0,22 R_f(CAF)=0,16 See fig:5
R_f was calculated according to the following equation [9] = distance traveled by the analyte / distance traveled by the solvent front.
As shown from the results, the greatest difference between R_f of the two drugs was obvious in experiment A, so it is considered the best mobile phase for separation of selected drugs

CONCLUSION

It was found that system (A) has better resolution than system B and C. The best solvent for separation of (CAF) and (PAR) from these three solvents is n-hexane-ethyl acetate-ethanol (2.5 + 1.5 + 0.4, v/v/v).
Limitation
Some other mobile phases were mentioned in the literature but not tried because of some solvent shortage and limited time of the project. No quantitative analysis was done because of in availability of densitometer.

FUTURE RESEARCH

Different stationary phase should be tried for the same mobile phase for example RP-TLC plates.

REFERENCES

1. Book: Harry W. Lewis and Christopher J. Moody. Experimental Organic Chemistry: Principles and Practice. Illustrated. Wiley Black well. 159–173. 978-0-632-02017-1. 13 Jun 1989.
2. Book: Tatchell, B.S. Furnis, A.J. Hannaford, and P.W.G. Smith, Vogel's Textbook of Practical Organic Chemistry. 5th. A.I. Vogel, A.R.. 0-582-46236-3.
3. Book: Reich, E.; Schibli A. High-performance thin-layer chromatography for the analysis of medicinal plants. Illustrated. Thieme. New York. 2007. 3-13-141601-7.
4. Budavaried, S.; The Merck Index: An Encyclopedia of Chemicals, Drugs and Biologicals, 14th ed., Merck & Co. Inc., Whitehouse Station, NJ, USA (2006).
5. K. Profitt (Ed.); Martindale–the Extra Pharmacopoeia: the Complete Drug Reference, 35th ed. Royal Pharmaceutical Society, London, UK (2006).
6. Tavallali, Hossein, S. F. Zareiyan, and Maryam Naghian. "An efficient and simultaneous analysis of caffeine and paracetamol in pharmaceutical formulations using TLC with a fluorescence plate reader." *Journal of AOAC International*. 2010; 94.4, 1094-1099.
7. Caitlin Sullivan and Joseph Sherma" Development and Validation of an HPTLC Densitometry Method for Assay of Caffeine and Acetaminophen in Multicomponent Extra Strength Analgesic Tablets", Journal Of Liquid Chromatography & Related Technologies. 2003; Vol. 26, Iss. 20.
8. Soponar, Florin, Augustin Cătălin Moţ, and Costel Sârbu. "Quantitative evaluation of paracetamol and caffeine from pharmaceutical preparations using image analysis and RP-TLC." Chromatographia. 2009; 69.1-2, 151-155.
9. HPTLC Fingerprint Analysis: A Quality Control for Authentication of Herbal Phytochemicals Article · January 2011 DOI: 10.1007/978-3-642-14025-9_7 Editor ManMohan Srivastava.