



Analytical Study for the Adsorption of Trifluoperazine HCl from Aqueous Solutions using inert Polyacrylic acid

Mohammad J.Hamzah *, Rawa M.M Taqi**, AbdulbariM.Mahood***

*Pharmaceutical chemistry department, Pharmacy College, Al-Nahrain University, Iraq.

**Pharmaceutical chemistry department, Pharmacy College, Al-Nahrain University, Iraq.

*** Pharmaceutical chemistry department, Pharmacy College, Kerbala University, Iraq.

*Corresponding author's E-mail: mohammedlord2003@yahoo.com

Accepted on: 24-09-2016; Finalized on: 30-11-2016.

ABSTRACT

The adsorption of trifluoperazine HCl on polyacrylic acid has been investigated in a batch adsorption process. The adsorption of trifluoperazine HCl was found to be dependent on pH, initial concentration, adsorbent dose and contact time. The result indicated that, optimum contact time was 12 hours, the adsorbent dose were 0.1545-0.1841 g of gel bead, pH=6.3 and the initial concentration of drug (70-100 mg.L⁻¹). The Langmuir, Freundlich and Temkin isotherm models were analyzed for the equilibrium adsorption data and the various isotherm parameters were evaluated. The adsorption mechanism was found to be followed the Freundlich model based on correlation coefficient (R²=0.9589). Therefore, polyacrylic acid can be used successfully as an alternative as adsorbent for trifluoperazine HCl.

Keywords: Adsorption, Trifluoperazine HCl, gel beads.

INTRODUCTION

Trifluoperazine is chemically 10-[3-(4-methylpiperazine-1-yl)propyl]-2-(trifluoromethyl)-10H-phenothiazine as shown in figure 1. Its molecular weight is 407.496 g/mol with an empirical formula C₂₁H₂₄F₃N₃S¹. It acts as an anti-psychotic agent, it blocks postsynaptic mesolimbic dopaminergic D1 and D2 receptors in the brain; depresses the release of hypothalamic and hypophysal hormones and is believed to depress the reticular activating system thus affecting basal metabolism, body temperature, wakefulness, vasomotor tone, emesis² and also it acts as anti-emetic, dopamine antagonist, antipsychotic³⁻⁷.

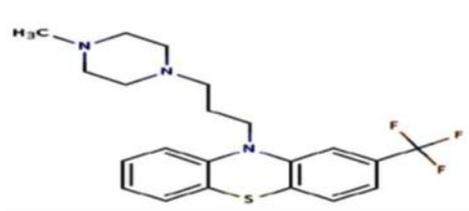


Figure 1: Chemical structure of trifluoperazine HCl.

Pharmaceuticals (antibiotics, anticonvulsants, antipyretics drugs, hormones) have recently been detected in sewage effluents (surface and ground water)⁸, and sometimes even in drinking water⁹⁻¹⁰, suggesting that their possible environmental impact is an emerging environmental issue¹¹⁻¹³.

Hydro gels such as polyacrylic acid and polyacrylic amide (are polymeric matrixes that swell but don't dissolve in the short term) in water. The swelling properties are due to the high thermodynamically affinity that this class of materials has for the solvent itself. In the past years this characteristic, coupled with a high versatility and a high

tunability of materials properties, lead to deep research and exploitation of hydrogels. These networks establish equilibrium with the liquid and temperature of their surroundings for shape and mechanical strength. Variations in the concentration, structure and/or functionality of the monomer and/or cross-linker used in such gels can change the structure. Indeed, many new gel-form materials, with a plethora of aims were developed and tested in different fields of engineering (e.g. environmental, electronics, biomedical), biotechnology and other disciplines¹⁴⁻¹⁹.

Research is being carried out for removal dyes and pharmaceutical compounds using chemical, physicochemical and biological treatment technologies, such as cloud point extraction, oxidation processes, coagulation flocculation, and adsorption have been used for the removal from waste water²⁰⁻²². However, adsorption is considered more effective and less expensive than other technologies. In the present study, detailed are carried out to remove and study of trifluoperazine HCl by adsorption technique using polyacrylic acid as an adsorbent. Various parameters affecting adsorption process, such as contact time, initial drug concentration, pH and adsorbent dose were investigated. In addition, analytical parameters were also calculated to determine adsorption mechanism was fitted into adsorption isotherms in order to give the best fit correlation.

MATERIALS AND METHODS

Apparatus

pH meter: Ametrohm E.632 pH meter (Switzerland) fitted with an Ametrohm combined glass electrode was

calibrated according to conventional methods and used to adjust the pH of the solution in each experiment.

Four digit electronic balances: Electronic balances Sartorius CE (Germany) four digits were used throughout this research work also Sartorius BL 210 S (Germany), max. 210 g, D 0.1 mg weighing was used.

Calipervernier: A Vernier caliper with 0.01 mm measuring accuracy was used for measurement of the diameter of the polyacrylic acid gel beads.

UV-Visible spectrophotometer: a double beam UV-Vis spectrophotometer shimadzu (Japan) was used for scan the spectrum and determination of drug concentrations.

Scanning electron microscopy (SEM): A scan electron microscope shimadzu was used for investigation the surface of polymer before and after the adsorption of trifluoperazine HCl.

Chemicals:

Polyacrylic acid: A commercial poly acrylic acid (PAA) hydrogel beads have (3.56 mm diameter and 0.0346 g weight) were used in this work.

Trifluoperazine Hydrochloride stock solution (500 $\mu\text{g}\cdot\text{mL}^{-1}$): A 500 $\mu\text{g}\cdot\text{mL}^{-1}$ stock solution of Trifluoperazine Hydrochloride was prepared by weighing 0.5g dissolved in distilled water and then diluted to the mark in 1000 mL volumetric flask. A series of working solutions of 20, 30, 40, 50, 70, 100, 150, and 250 $\mu\text{g}\cdot\text{mL}^{-1}$ were prepared by dilution adequate volumes of stock solution with distilled water.

Hydrochloric acid (HCl), approximately 1N and 0.1N:

A stock solution of HCl (1N) was prepared by transferring 8.4 mL of concentrated acid (11.9N) into 100 mL volumetric flask and diluted to the mark with distilled water. Then 10 mL of stock solution of hydrochloric acid was diluted to the mark with distilled water to prepare 0.1N.

Sodium hydroxide (NaOH), 0.1N: a 2.0g of sodium hydroxide was weighed in a weighed plat and transferred into 500 mL volumetric flask. The volume was made up to 500 mL with distilled water. The solution was stored in a plastic bottle.

Perliminary Investigations

The development of adsorption methods for the removal of drugs has increased considerable in recent years because of their importance in pharmaceutical industries and pharmaceutical analysis. Therefore, the interaction between the adsorbate polyacrylic acid hydrogel and the adsorbent trifluoperazine HCl was study. Polyacrylic acid gel bead was immersed into trifluoperazine HCl solution for period of time. The morphological structure of adsorpante was characterized by electron scanning microscope (SEM) and shown in Figure 2 (a, b, c, d). It is clearly show the difference between the surface of gel bead before and after the adsorption of trifluoperazine HCl. Based on this phenomenon polyacrylic acid gel bead was used as adsorbate bead for trifluoperazine HCl from aqueous solutions.

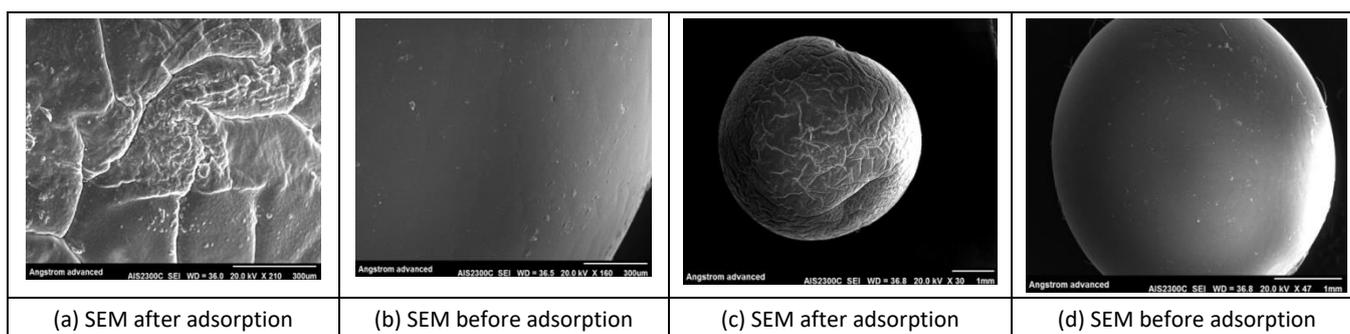


Figure 2: a,c shows the electron scanning microscope(SEM) after adsorption of drug into it surface, while b,d shows SEM before adsorption.

The maximum absorbance of the trifluoperazine HCl was confirmed by scanning the drug standard solution over the spectral range of 180-350nm by using UV-VIS spectrophotometer. The absorbance of those standard samples was measured at the corresponding maximum wavelength ($\lambda_{\text{max}}=258\text{nm}$) as shown in figure 3.

Showing the maximum absorbance at 258nm. To determine the remaining and removal trifluoperazine HCl concentration by polyacrylic acid, a calibration curve was first obtained from a series of predetermined concentration of drug standard solutions as shown figure 4.

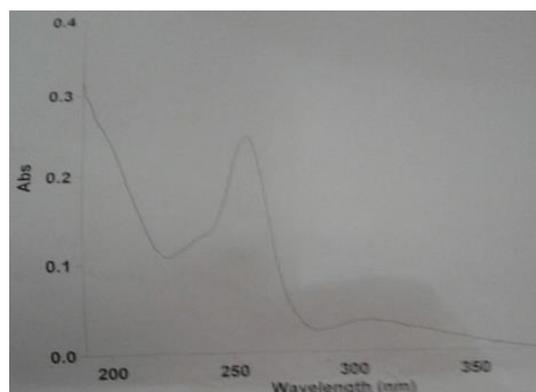


Figure 3: Spectrum of trifluoperazine HCl was measured against blank solution distilled water

The results obtained showed that the rate of adsorbed trifluoperazine HCl onto gel bead was rapid and then became slower near the equilibrium.

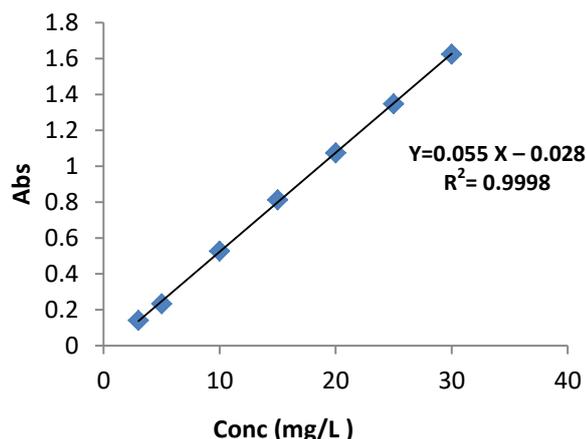


Figure 4: Calibration curve of trifluoperazine HCl.

The capacities at each time contact were evaluated using the following equation [22].

$$Q_e = (C_o - C_e) V / m$$

Where **Q** is the amount of drug adsorbed at time *t* or at equilibrium (mg / g). **C_o** and **C_e** are the initial and final concentration (concentration at *t* time or at equilibrium) of drug (mgL⁻¹), **V** is the volume of drug solution used (Liter), and **m** is the weight of the hydrogel bead used (gram). Most of the maximum quantity adsorption of trifluoperazine HCl was attained after about 12 hours of contact time. The results indicated that the fact that a large number of vacant surface site were available for adsorption during the initial stage near the equilibrium the remaining vacant surface sites were difficult to be occupied due to the slow pore diffusion of the solute molecules on the solid and the bulk phase.

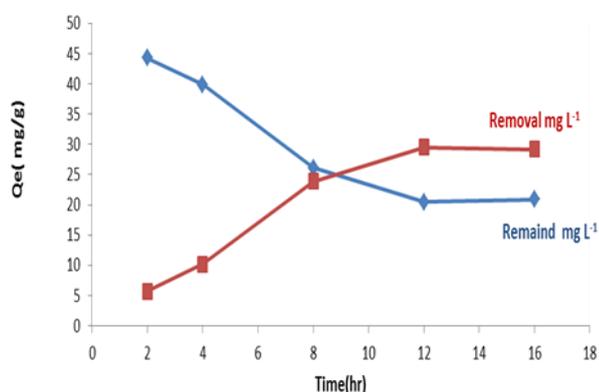


Figure 5: Effect of contact time between trifluoperazine and polyacrylic acid.

Experiments were conducted varying the solution pH from 3 to 8, while the remain of the factors were kept constant. The maximum removal of trifluoperazine HCl was observed at pH =6.3. The adsorption mechanisms on the polyacrylic acid surface reflect the nature of physiochemical interaction of the solution.

Effect of Adsorbent Dose

The effect of adsorbent dose is an important parameter because this determines the capacity of adsorption for trifluoperazine HCl initial concentration. The adsorption efficiency of trifluoperazine HCl as a function of adsorbent dose was studied using different number of hydrogel bead (1 -5 gel beads) with different weight ranging from 0.0405, 0.0750, 0.1140, 0.1545 and 0.1841g weight, while The solutions were adjusted at pH of 6 and lifted for 12 hour at room temperature. The residual drug concentration was determined using UV-Visible spectrophotometer and the percentages of drug removal were then evaluated. The obtained results indicated that the removal percentage of trifluoperazine HCl adsorption slightly increases with the adsorbent dose loading up to 4-5 gel beads (0.1545-0.1841g). This result can be explained by the fact that the adsorption sites remain unsaturated during the adsorption interaction where as the number of a sites available for adsorption site increases by increasing the adsorbent dose.

Effect of initial concentration of Trifluoperazine HCl

Adsorption equilibrium was determined using different initial concentrations from trifluoperazine HCl. A 25ml solutions ranging (20-100mg.L⁻¹) from trifluoperazine HCl were used and adjusting the other variables. The residual and percentage removal or adsorbed drug was determined from the standard curve as in previous section. The obtained results indicated that the percentage removal or adsorbed by polyacrylic acid increases with increasing the initial drug concentration up to (70-100mg.L⁻¹).

Adsorption isotherms

Different adsorption isotherms were used such as Langmuir Adsorption Isotherm, Freundlich Isotherm and Temkin isotherm to study. The adsorption mechanism and relationship between the concentration of the adsorbate and adsorption capacity of both the adsorbent.

Langmuir model: $C_e / Q_e = 1 / Q_{max} K_L + C_e / Q_{max}$

Freundlich model: $\log Q_e = \log K_f + 1/n \log C_e$

Temkin model: $Q_e = RT/b \ln A + RT/b \ln C_e$

Where **Q_e** is the amount of trifluoperazine HCl adsorbed at equilibrium (mg/g), **C_e** is the equilibrium concentration of trifluoperazine HCl solution (mg.L⁻¹), **Q_{max}** is the maximum capacity of adsorption (mg/g) and **K_L** is the Langmuir adsorption constant (L/g) and is related to the free energy of adsorption, **K_f**(ml/g) and **n** are the Freundlich adsorption isotherm constants, being indicative of the adsorption extent and the degree of nonlinearity between solution concentration and adsorption, respectively. The **b** and **A** were calculated from slope and the intercept of the linear plot of **Q_e** versus **lnC_e** shows that adsorption follows Temkin isotherm. Where **A** (L/g) is the equilibrium binding

constant corresponding to the maximum binding energy and constant **b** is related to the heat of sorption (298K). The obtained results were plotted for each model as shown in figures 6, 7, 8. In view of the value of linear regression coefficients (R^2), it was found that the adsorption process for the drug is well described and more fitted with Freundlich ($R^2 = 0.9589$) rather than Langmuir and Temkin isotherms.

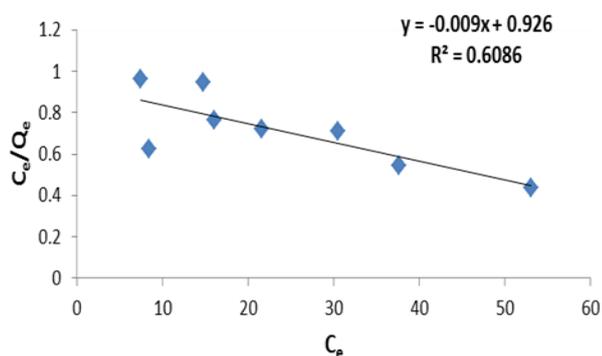


Figure 6: Langmuir model shows that $R^2 = 0.6086$

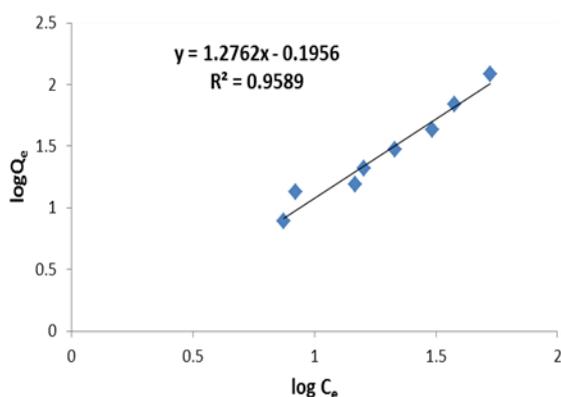


Figure 7: Freundlich model shows $R^2 = 0.9589$

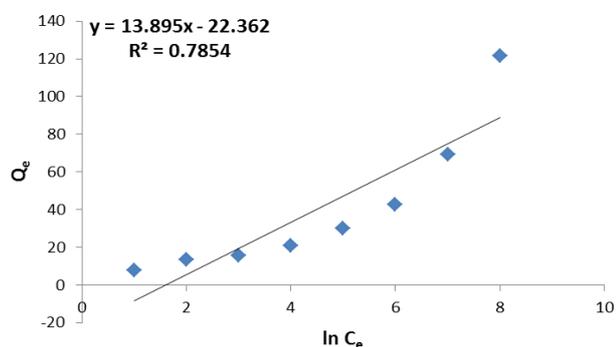


Figure 8: Temkin model shows $R^2 = 0.7854$

CONCLUSIONS

In present study polyacrylic acid hydrogel beads was selected as a local, cheap and readily available adsorbent for the removal and adsorption of trifluoperazine HCl from aqueous solution. The gel bead was studied by batch adsorption experiments. The adsorption of trifluoperazine HCl was found to be dependent, pH, Initial concentration, adsorbent dose and contact time. The Langmuir, Freundlich and Temkin

isotherm models were analyzed, adsorption data and the various isotherm parameters were evaluated. Finally once we can conclude that the inert polymer of gel bead can be used successfully for adsorption and removal of trifluoperazine HCl and the other toxic compounds from aqueous solutions.

REFERENCES

1. Ahmed N.R, "Ultraviolet Spectrophotometric Determination of Trifluoperazine. HCl in Pharmaceutical Preparations and Environmental Wastewater Samples: Application to Content Uniformity Testing", *Journal of Pharmaceutical Analysis*, 3, 2014, 30-34.
2. The Pharmaceutical Codex, London, 1979, 961.
3. Khammas. Z.A.A and Rashid.R.A, "Mutual Determination of Trifluoperazine Hydrochloride and Vanadium (V) Ions in Real Matrices by Visible Spectrophotometry after Cloud Point Extraction", *Science Journal of Analytical Chemistry*, 3, 2015, 61-70.
4. Al-Rufaie.M.M and Kathem.K.H, "NEW SPECTROPHOTOMETRIC METHOD FOR DETERMINATION TRIFLUOPERAZINE HYDROCHLORIDE IN PHARMACEUTICAL PREPARATIONS BY USING OXIDATIVE COUPLING REACTION", *World Journal of Pharmaceutical Research*, 3, 2014, 1202-1214.
5. Sharma.M.C and Sharma.S, "Development and validation of densitometry estimation of trifluoperazine hydrochloride in dosage form", *American-Eurasian journal toxicology sciences*, 3, 2011, 101-104.
6. QASIM.A.W and KHAMMAS.Z.A.A, "An Indirect Atomic Absorption Spectrometric
 - a. Determination of Trifluoperazine Hydrochloride in Pharmaceutical Formulations Based on Chelate Formation with Palladium (II)", *E-Journal of Chemistry*, 7, 2010, 433-441.
7. David.S.B and Polkinghorn, "Evidence-based pharmacotherapy of generalized anxiety disorder", *International Journal of Neuropsychopharmacology*, 8, 2005, 293-302.
8. Mohannad. Q, Mustafa. K, Fida .M, Shlomo. N, Sabino A.B.A, Laura.S and Rafik. K, "Stability and Removal of Naproxen and Its Metabolite by Advanced Membrane Wastewater Treatment Plant and Micelle-Clay Complex", *Clean – Soil, Air, Water*, 41, 2013, 1-7.
9. Carballa. M, Omil. F, Lema. J .M, Llopart. M, Garcia-Jares. C, I.Rodriguez. I and Gomez. M, et al, "Behavior of Pharmaceuticals, Cosmetics and Hormones in a Sewage Treatment Plant", *Water Res*, 38, 2004, 2918-2926.
10. Kolpin.D.W, Furlong.E.T, Meyer.M.T, Thurman.E.M, Zaugg.S.D, Barber.L.D and Buxton.H.T, "Pharmaceuticals, Hormones and Other Organic Wastewater Contaminants in US Streams", *Environ. Sci. Technol*, 36, 2002, 1202-1211.
11. Stackelberg.P. E, Furlong. E. T, Meyer. M. T, Zaugg. S. D, Henderson. A. K and Reissman.D.B, "Persistence of Pharmaceutical Compounds and Other Organic Wastewater Contaminants in a Conventional Drinking-

- Water Treatment Plant", *Sci. Total Environ*, 329, 2004, 99–113.
12. Daughton. C. G, "Non-Regulated Water Contaminants: Emerging Research", *Environ. Impact Assess. Rev*, 24, 2004, 711–732.
 13. Fent. K, Weston. A. A and Caminada. D, "Ecotoxicology of Human Pharmaceuticals", *Aquatic Toxicol*, 76, 2006, 122–159.
 14. Chirani.N, Yahia. LH and Gritsch .L, "History and Applications of Hydrogels", *J Biomedical Sci*, 4, 2016, 1-23.
 15. Lee. SC, Kwon. IK and Park. K, "Hydrogels for delivery of bioactive agents: a historical perspective", *Adv Drug Deliv Rev* 65, 2013, 17-20.
 16. Wei .Y S and Charlotte A.E, "Short to ultrashort peptide hydrogels for biomedical uses", *Materials Today*, 17, 2014, 381-388.
 17. Buwalda. SJ, Boere. KW, Dijkstra. PJ, Feijen. J, Vermonden. T and Hennink. WE, " Hydrogels in a historical perspective: from simple networks to smart materials", *J Control Release*, 190, 2014, 254-273.
 18. Yom-Tov. O, Neufeld. L, Seliktar. D and Bianco-Peled. H, "A novel design of injectable porous hydrogels with in situ pore formation" , *Acta Biomater* 10, 2014, 4236-4246.
 19. Juergen. S, Ronald.S and Michael. R, "Fundamental and applications of controlled release drug delivery", Springer, New York NY, 2012, 75-106.
 20. Neelmegani. R, Baskaran. V, Dhancekar. R and Viruthgiri, T, "Decolorisation of synthetic dyes using rice straw attached pleurotusolereths", *Indian. J chem. Techo*, 11, 2004, 622-625.
 21. LiLi. L and Liping GUO, "Adsorption of congo red from aqueous solution on to Ca-bentonite", *Journal of Hazardous Materials*, 161, 2009, 126-131.
 22. Ahmed. M S and Mohammad. J. H , " New approach for removal of total hardness (Ca²⁺, Mg²⁺) from water using commercial polyacrylic acid hydrogel beads, study and application", *International journal of Advanced Biological and Biomedical Research*, 1, 2013, 1142-1156.

Source of Support: Nil, **Conflict of Interest:** None.