Development and Validation of a Reverse Phase-HPLC method for Methylphenidate and its disposal using activated charcoal based system.

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INTRODUCTION:

- According to the survey conducted by National Institute for drug abuse, in 2009 over 7 million Americans used prescription drugs nonmedically and there were about 1000 reports of occurrence of pharmaceuticals in sewage and ground waters.
- FDA recommends proper disposal of unused prescribed medication by various ways, since improper disposal can lead to safety, and environmental hazards.
- Deterra™ drug deactivation system offers a unique disposal method to deactivate unused, residual or expired medications by using granular activated carbon within a pouch in a convenient, effective and safe manner.
- In this study, a robust and validated method for methylphenidate was developed using high-performance liquid chromatography (HPLC) and the deactivation efficiency was tested by using this deactivation system.

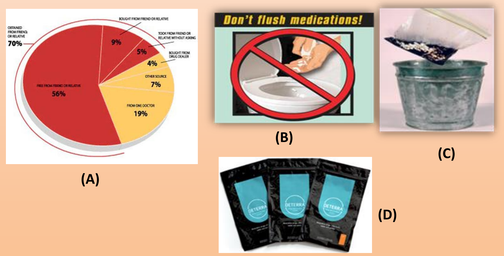


Figure.1: A) Survey showing abuse of prescription medications. B) Environmental safety concern with drug disposal of prescription medications. C) Traditional drug disposal methods. D) Deterra™ Drug Deactivation system.

METHODS

1) HPLC method development and validation for Methylphenidate was performed using following parameters:

HPLC Parameters	Methylphenidate
Column	Kinetex 5u, Biphenyl 100A, 250* 4.6mm
Mobile Phase (%)	50:50 (v/v), Methanol (0.1% FA): water 0.1% FA, pH 6.8
Flow Rate (ml/min)	1.0
Injection Volume (µl)	20
UV detector (nm)	258
Retention time (min)	~9.0

Table.1: HPLC parameters for detection on Methylphenidate by reverse phase chromatography.

2) Adsorption study for deactivation of Methylphenidate:

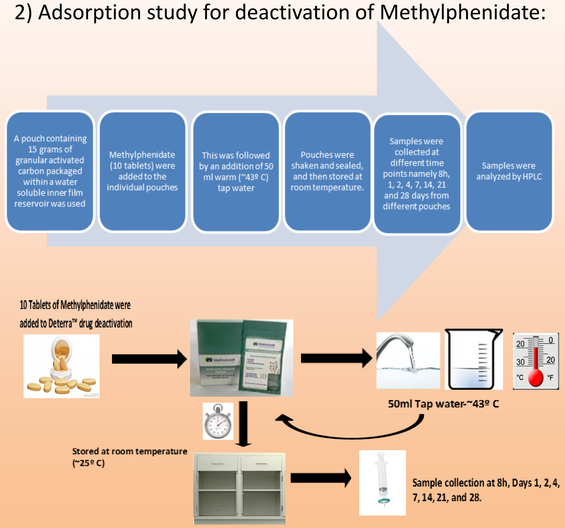
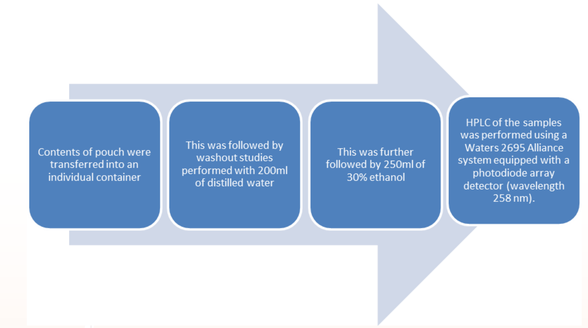


Figure.2 : Protocol- Rate and extent of adsorption for Methylphenidate

3) Deabsorption study for washout from water and ethanol:



After 28 days, any potential leaching of the drug adsorbed to activated carbon was examined.

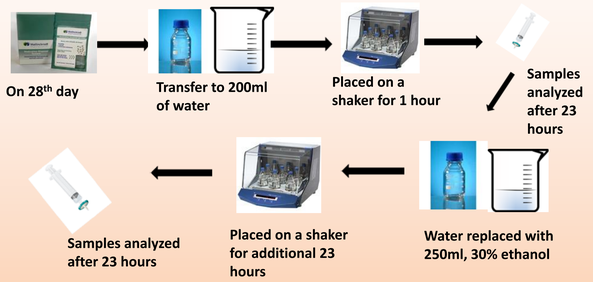


Figure.3: Desorption studies with water followed by ethanol.

RESULTS

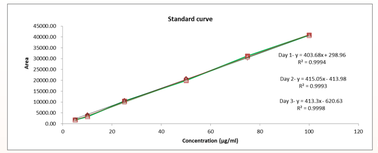
Linearity was established over the concentration range of 5-100 µg/mL with a correlation coefficient ≥ 0.999. The limits of detection and quantitation were 1.38 and 4.17 µg/mL, respectively. The intraday and inter day variation was found to be within 1-6%. In case of the deactivation study, samples tested at the 8 hour time-point already showed more than 98% of drug deactivation and after 24 hours, 100% of the drug was deactivated.

Concentration (µg/ml)	Average	SD	Accuracy (%)	Precision (%)
50	48.59	2.09	97.17	4.31
75	73.05	2.92	97.40	4.00
100	94.56	5.70	94.56	6.03

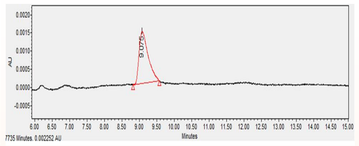
Table.2: Intraday accuracy and precision

Concentration (µg/ml)	Average	SD	Accuracy (%)	Precision (%)
50	49.33	1.76	98.66	3.57
75	74.11	2.68	98.81	3.61
100	97.10	4.76	97.10	4.90

Table.3: Interday accuracy and precision

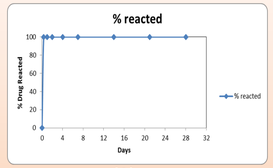


(A)



(B)

Figure 4: A) Standard curve plot of Methylphenidate on three different days. B) Chromatogram representation of Methylphenidate at ~9.0 minutes.



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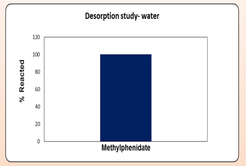

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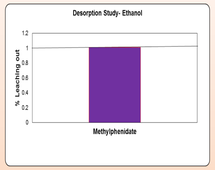

(C)

Figure.5: A) % deactivation of Methylphenidate with activated carbon over a period of 28 days. Desorption study of Methylphenidate: B) Washout in water C) Washout in ethanol.

CONCLUSION

A sensitive, precise and robust HPLC method was developed for the detection of methylphenidate. Based on this method, the efficiency of an activated carbon based deactivation system was tested with successful adsorption of methylphenidate as a model psychoactive medication.

Acknowledgment:

This project was funded by Verde Technologies (Minnetonka, MN) as a SBIR Phase II Contract from NIDA

Reference

1. Herwadkar A, Singh N, Anderson C, Korey A, Fowler W, Banga AK. Development of disposal systems for deactivation of unused/residual/expired medications. Pharm Res. 2015 Aug 12. [Epub ahead of print]