



RESEARCH ARTICLE

Development and validation of simple UV-spectrophotometric method for the estimation of polystyrene plastic/microplastic

Tanish Goyal¹, Ghanshyam Das Gupta², Sant Kumar Verma³

¹Department of Pharmaceutical Analysis, ISF College of Pharmacy, Moga, Punjab, India, ²Department of Pharmaceutics, ISF College of Pharmacy, Moga, Punjab, India, ³Department of Pharmaceutical Chemistry, ISF College of Pharmacy, Moga, Punjab, India

ABSTRACT

Polystyrene is a widely used plastic for household purposes or in industrial packaging. However, polystyrene can be categorized as a potent carcinogenic due to its monomer unit. Polystyrene comprises several styrene units that easily leach as styrene microplastics on contact with hot or fatty material. A ultraviolet (UV) spectrophotometer method has been developed for the determination of polystyrene. Polystyrene in an aqueous solution with tetrahydrofuran was estimated at a wavelength ranging from 200 to 300 nm. The $\lambda_{\max}$ of polystyrene was found to be 261.5 nm. The method was linear for the polystyrene concentration ranging from 20 to 120 $\mu\text{g/mL}$. The limit of detection was founded to be 1.65 $\mu\text{g/mL}$, and the limit of quantification was 5 $\mu\text{g/mL}$. Interday and intraday precision were performed for the developed method, and the RSD was <2%. Further, the developed method was evaluated with other International Conference of Harmonization parameters, such as ruggedness and robustness, showing positive results. The developed UV spectrophotometric method would be useful for estimating polystyrene plastic or microplastic in polluted water in a cost and time-efficient manner.

KEY WORDS: Microplastic, Polystyrene, Ultraviolet spectrophotometer method, White pollution

INTRODUCTION

Plastic is widely used around the globe due to its low cost and ease of processibility; however, the downside of its use is its non-biodegradability which leads to white pollution.^[1] Furthermore, because of polystyrene's non-biodegradable nature and chemical stability, it is usually disposed of by incineration, which releases toxic byproducts. Furthermore, catalytic or thermal degradation of waste polystyrene plastic into fuel oils has been explored recently.^[1-3] Among various plastic used daily, polystyrene is not easily degraded and is the major contributor to white pollution. Polystyrene is a plastic made up of styrene (a monomer unit of polystyrene). The number of households is made up of polystyrene, such as plastic utensils, disposable cups

and plates, coffee cup lids, egg cartons, cafeteria trays, take-out containers, desk accessories, toys, video cassettes and cases, clamshell containers, packaging peanuts, and insulation board and other expanded polystyrene products (e.g., Styrofoam).^[4] Industries use polystyrene in various forms, such as copolymerized for synthesizing different plastics, soft form for insulating purposes, and hard form for housing casing products.^[5] Household plastic leaches styrene in the form of microplastic, a human carcinogen, into food when they contact with heated or fatty or acidic foods.^[4] Microplastics are simpler and tiny particles of

Address for Correspondence:

Sant Kumar Verma

E-mail: santkumarverma19@gmail.com

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plastic, mainly resulted due to breakdown of larger plastics. It have been reported that these microplastic are uptaken by various aquatic and terrestrial species that lead to ingestion problem, gastric problem, and ever death of the organism.^[6] Furthermore, polystyrene reaches water bodies through plastic manufacturers, distributors, and household waste. A recent survey reported that nearly 2.7×10^6 metric ton was present in the ocean worldwide as Styrofoam, among which about 20% were degraded.^[7]

In this research, simple attempts were made to develop a simple, accurate and precise ultraviolet (UV) spectrophotometer method for determining polystyrene. Furthermore, the developed method was validated according to International Conference of Harmonization (ICH) guidelines.^[8]

MATERIALS AND METHODS

Materials

Polystyrene was obtained from Sigma- Aldrich Chemical Pvt. Ltd has an average molecular weight of 280,000. Tetrahydrofuran (THF; 99.5% pure) was received from Loba Chemie Pvt. Ltd. and used as a diluent.

Preparation of stock solution and sample solution

The stock solution was prepared by dissolving 10 mg polystyrene in a 10 mL THF in a 10 mL volumetric flask, resulting in 1000 $\mu\text{g/mL}$ solution. For the preparation of sample solution ranging from concentration 20 to 120 $\mu\text{g/mL}$ (20, 40, 60, 80, 100, and 120 $\mu\text{g/mL}$), 0.2, 0.4, 0.6, 0.8, 1, and 1.2 mL were taken from the stock solution and made up the volume up to 10 mL with THF in a 10 mL volumetric flask.

RESULTS AND DISCUSSION

Determination of λ_{max} of polystyrene

For peak identification, 0.2 mL of stock solution was diluted up to 10 mL THF in a 10 mL volumetric flask to prepare a reference solution. Further, a UV spectrophotometer was used to scan the polystyrene from 200 nm to 300 nm to determine the λ_{max} of polystyrene. Diluent was used as blank. The UV spectrum of polystyrene was taken in THF and is presented in Figure 1. The λ_{max} of polystyrene was found to be 261.5 nm. The obtained spectrum was matched with the reference spectrum of polystyrene.

Linearity

For the linearity studies, six solutions of concentration ranging from 20 to 120 $\mu\text{g/mL}$ (20, 40, 60, 80, 100, and

120 $\mu\text{g/mL}$) were prepared by taking 0.2, 0.4, 0.6, 0.8, 1.0, and 1.2 mL from the stock solution and diluting them up to 10 mL with THF in a 10 mL volumetric flask. The calibration curve for polystyrene in THF was obtained using the 20–120 $\mu\text{g/mL}$ solution [Table 1]. The calibration curve [Figure 2] was drawn with the regression equation $y = 0.0028x - 0.0077$ and r^2 value 0.996, which showed good linearity [Table 1 and Figure 3].

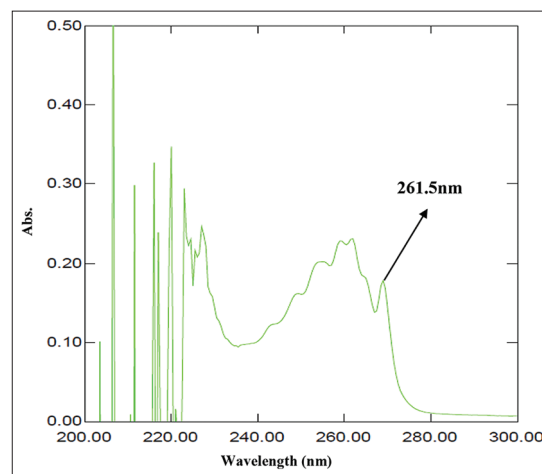


Figure 1: Ultraviolet spectrum (in tetrahydrofuran) showing λ_{max} of polystyrene

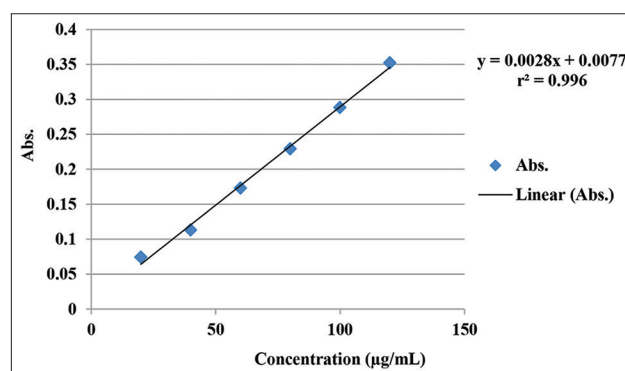


Figure 2: Standard calibration curve of polystyrene by ultraviolet

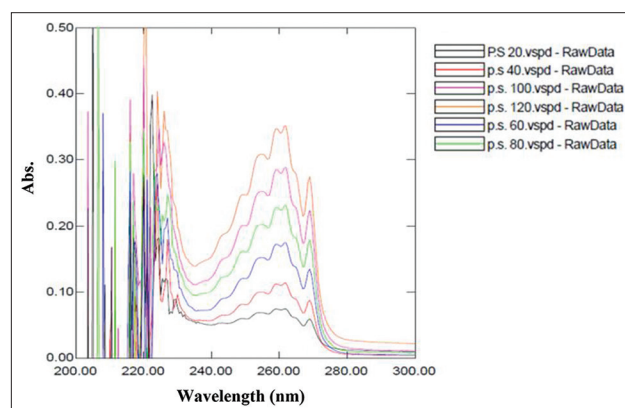


Figure 3: Overlay spectra of calibration curve for polystyrene in tetrahydrofuran

Table 1: Linearity data of polystyrene

Concentration (µg/mL)	Absorbance			Mean Abs	Standard Deviation (SD)
	Abs1	Abs2	Abs3		
20	0.074	0.077	0.072	0.074	0.002
40	0.113	0.114	0.113	0.113	0.0004
60	0.174	0.173	0.174	0.173	0.0004
80	0.231	0.227	0.231	0.229	0.001
100	0.289	0.286	0.290	0.288	0.001
120	0.351	0.354	0.353	0.352	0.001
Average SD					0.001

Table 2: Intraday precision data for polystyrene

Concentration (µg/mL)	Trial 1	Trial 2	Trial 3
80	0.234	0.231	0.233
80	0.232	0.233	0.230
80	0.232	0.234	0.235
80	0.232	0.233	0.231
80	0.231	0.233	0.232
80	0.231	0.234	0.229
Mean±SD	0.232±0.001	0.233±0.001	0.231±0.002
% RSD	0.4	0.4	0.8

Table 3: Interday precision data for polystyrene

Concentration (µg/mL)	Day 1	Day 2	Day 3
80	0.234	0.230	0.236
80	0.232	0.235	0.233
80	0.232	0.230	0.232
80	0.232	0.233	0.232
80	0.231	0.232	0.234
80	0.231	0.235	0.235
Mean±SD	0.232±0.001	0.232±0.002	0.233±0.001
% RSD	0.4	0.8	0.4

Limit of detection (LOD) and limit of quantification (LOQ)

The LOD is the lowest amount of analyte that can be detected but not quantitated as an exact value. The limit of quantitation is the lowest amount of analyte that can be quantitatively determined in a sample with suitable precision and accuracy. The LOD and LOQ for polystyrene were 1.65 µg/mL and 5 µg/mL, respectively.

Precision

The precision defines the closeness of agreement among a series of measurements obtained from multiple sampling of the same sample solution under the prescribed conditions. Precision may be considered at three levels: repeatability,

Table 4: Accuracy readings of polystyrene

Concentration (µg/mL)	Abs.	Recovered concentration (µg/mL)	Mean (µg/mL)	% Recovery
64	0.185	63.10	63.46	99.15
64	0.186	63.82		
64	0.187	63.46		
80	0.234	80.60	79.88	99.85
80	0.232	79.89		
80	0.230	79.17		
96	0.276	95.60	96.31	100.3
96	0.278	96.32		
96	0.280	97.03		

intermediate precision, and reproducibility. The precision of an analytical method should be explored using homogeneous and authentic samples. However, it can also be analyzed using an artificially prepared sample solution. The precision is commonly expressed as the standard deviation and relative standard deviation. The results of interday precision and intraday precision experiments are shown in Tables 2 and 3. The developed method was found to be precise as the % RSD values for intraday precision and interday precision was ≤2%, and it is according to the recommendation of ICH Q2(R1) guidelines.

Accuracy

The method's accuracy was determined in terms of % recovery of standard from solution. Recovery studies were carried out by the addition of polystyrene solution at the three concentration levels, that is, 80%, 100% and 120%, in pre-analyzed sample [Table 4].

Robustness

The robustness of the method was studied by changing the experimental conditions. A set of parameters, that is, wavelength (±1 nm), were altered. The standard deviation and relative standard deviation were calculated. The RSD of the developed method was found to be <2% [Table 5]. It was observed that

Table 5: Robustness data for change in wavelength

S. No.	Absorbance		
	260.5 nm	261.5 nm	262.5 nm
1	0.225	0.230	0.228
2	0.227	0.231	0.230
3	0.228	0.233	0.231
4	0.228	0.233	0.231
5	0.228	0.233	0.230
6	0.227	0.232	0.229
Mean±SD	0.227±0.001	0.232±0.001	0.230±0.001
RSD	0.60	0.43	0.43

Table 6: Ruggedness data for change in analyst

S. No.	Analyst	
	Analyst 1	Analyst 2
1	0.234	0.235
2	0.232	0.235
3	0.232	0.230
4	0.232	0.233
5	0.231	0.232
6	0.231	0.230
Mean±SD	0.232±0.001	0.232±0.002
RSD	0.4	0.8

there were no marked changes in the absorbance; these results demonstrate that the developed UV method is robust.

Ruggedness

A change in the analyst was made to carry out the ruggedness of the developed method. However, the concentration of the reference solution was 80 µg/mL prepared according to the dilution method used in linearity. The developed method showed <2% RSD, which signified good ruggedness [Table 6].

CONCLUSION

Obtained results of development and validation parameters demonstrate a rapid, precise, accurate, and robust UV spectrophotometric method for determining polystyrene. Therefore, this method would be useful to determine the polystyrene plastic or microplastic in various waste water without any interference from excipients.

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