Quantitative estimation of iron in Unani herbo-mineral products employing ion exchange chromatography

Tajuddin, S H Afaq * and Yusuf Salim
Department of Ilmul Advia
Faculty of Unani Medicine, Aligarh Muslim University, Aligarh – 202 002, India
*Correspondent author, E-mail: sh_afaq@rediffmail.com

Abstract

The ion exchange chromatography was conducted on Unani Syrup preparation of iron (Sharbat-e-faulad), manufactured by the three different pharmaceutical companies and compared with the standard Fesovit spansules. The ion exchanger was antimony (v) arsenosilicate, which is of crystalline nature and found to be highly selective for iron. The results indicate that the syrup manufactured by Hamdard (Wakf) Laboratories, Delhi (SFH) contains 96.11 ± 3.5 mg/10 ml. The sample of Rex (U&A) Remedies Pvt. Ltd., Delhi (SFR) contains 78.4 ± 1.5 mg/10 ml and Dwakhana Tibbia College, Aligarh (SFD) contains 30.37 ± 1.8 mg/10 ml iron. The Fesovit spansules (FS) manufactured by Glaxo-Smith Kline Pharmaceuticals Ltd., Bangalore contains 47.7 ± 0.9 mg per spansule. SFH comprises highest content of iron and most suitable for severe iron deficiency anaemia. SFR is the standard sample and negligible discrepancy noted in the content of iron claimed by manufacturer, whereas in SFD 25.308% less iron was determined in comparison to claimed amount of iron. Standard operational procedure (SOP) is required to be implemented for standard preparations of traditional medicines.

Keywords: Iron estimation, Ion exchange chromatography, Unani syrup preparation, Sharbat-e-faulad, Fesovit, Iron deficiency anaemia.

IPC code: Int. cl.7 — A61K 33/26, B01J 39/06

Introduction

Iron is an integral part of haemoglobin, myoglobin, cytochromes, catalases and certain other enzyme systems, which assist in the biochemical reactions in the cells. Nearly half of the enzymes of the Kreb's cycle require iron. It is an important element of the electron carriers, which are essential for most of the oxidation reactions in human cells. Iron deficiency directly affects the body's immune system, functioning of the brain and overall development. A survey carried out by WHO shows that 50% of children and women, 25% of men in developing countries and 7-12% in developed countries are iron deficient (DeMaeyer & Adiels-Tegman, 1985). The iron deficiency develops as a result of increased requirement, decreased intake, decreased absorption of iron, and/or regular blood loss (Harrison, 1987). The simplest, cheapest and most preferred treatment of iron deficiency is iron supplemented by oral administration. The maximum content of iron required for this purpose is 180 – 200 mg/day in three divided doses (Gilman et al., 1992; Harrison, 1987). Keeping these facts in consideration a study was designed to estimate the total iron content in Sharbat-e-faulad which is an important Unani syrup preparation.
herbo-mineral formulation being extensively prescribed for anaemia in the system.

**Methodology**

The market samples of *Sharbat-e-faulad* and Fesovit were procured from three Unani manufacturing houses:

(i) Hamdard (Wakf) Laboratories, New Delhi (SFH), (Batch No. 60): Each 10 ml of the syrup contain Burada Faulad (iron dust) 72 mg, Sammulfar (arsenic) 3.6 mg, Sugar syrup q.s.

(ii) Rex (U & A) Remedies (Pvt.) Ltd., New Delhi (SFR), (Batch No. 63/18): Each 10 ml of the syrup contain Burada Faulad (iron dust) 80 mg, Aab-e-Anar Tursh (juice of *Punica granatum* Linn.) 2 ml, Qand Safaid (sugar) 8 mg.

(iii) Dawakhana Tibbia College, AMU, Aligarh (SFD), (Batch No. 35.36): Each 10 ml of the syrup contain Burada Faulad (iron dust) 120 mg, Tursh Shora (HNO₃) 0.3 ml, Tursha Namak-e-Shor (HCl) 0.1 ml, Tursha Kibrit (H₂SO₄) 0.1 ml, Sirka Jamun [vinegar of *Syzygium cumini* (Linn.) Skeels syn. *Eugenia jambolana* Lam.] 3 ml, Sat Lemoon (Citric acid) 60 mg, Tukhm Karafs (fruits of *Apium graveolens* Linn.) 6 mg, Badian (fruits of *Foeniculum vulgare, Mill.*) 3 mg, Ajwain Desi [fruits of *Trachyspermum ammi* (Linn.) Sprague] 3 mg, Saatar Farsi (whole plant of *Zataria multiflora Boiss.*) 3 mg, Anisoon Roomi (fruits of *Pimpinella anisum* Linn.) 3 mg, Hilteet (resin of *Ferula foetida* Regel) 3 mg, Kashniz Khushk (Fruits of *Coriandrum sativum* Linn.) 3 mg, Filfil Daraz (fruits of *Piper longum* Linn.) 3 mg, Kumdr Gond (oleo-gum-resin of *Boswellia serrata* Roxb.) 3 mg, Saad Koofi (tubers of *Cyperus rotundus* Linn.) 3 mg, Darchini (bark of *Cinnamomum zeylanicum* Blume) 3 mg, Khurfa Siyah (aerial part of *Portulaca oleracea* Linn.) 3 mg, Joz Bua (roots of *Valeriana officinalis* Linn.) 3 mg, Tukhm Halon (seeds of *Lepidium sativum* Linn.) 3 mg, Tukhm Piaz (seeds of *Allium cepa* Linn.) 3 mg, Zanjabeel Khushk (rhizome of *Zingiber officinale* Rosc.) 3 mg, Filfil Siyah (fruits of *Piper nigrum* Linn.) 3 mg, Zeera Safaid (fruits of *Cumimum cyminum* Linn.) 1.5 mg, Tursha Sammulfar (Arsenous acid) 0.2 ml, Qand Safaid (sugar) q.s.

(iv) Fesovit Spansule (FS) manufactured by Glaxo-Smith Kline Pharmaceuticals Ltd., Bangalore (Batch No. 2006): Each spansule contains dried Ferrous sulphate 150 mg, Folic acid 1mg, Cyanocobalamin 15 mcg, Pyridoxin hydrochloride 2 mg and Nicotinamide 50 mg. The spansule was taken for a comparative study.

Estimation of total iron content in different market samples of *Sharbat-e-faulad* and Fesovit spansule was carried out by the method of ion exchange chromatography using Antimony (v) arsenosilicate as exchanger as described by Varshney et al (1994a). Varshney et al (1994a) reported that the Na⁺ ion exchange capacity (1.62 mcq/dry gr) and the distribution coefficient (Kd) studies for various metals for Antimony (v) arsenosilicate. After comparing it with other exchanger they suggested that Antimony (v) arsenosilicate possesses high selectivity for iron (III) and have reasonably good thermal and chemical stability in addition to being crystalline (Varshney et al, 1994b).

For stock solution 5 ml of each sample of *Sharbat-e-faulad* (SF), was
heated with a 10 ml of the oxidizing mixture (conc. HNO$_3$ + HClO$_4$ in 1: 2 volume ratios) to destroy the organic matter completely till a clear solution was obtained. The volume of this solution was reduced to 1-2 ml by continuous heating to remove excess acid and after cooling the volume was maintained to 100 ml with de-mineralized water (DMW) in a standard volumetric flask. Similarly, one spansule of Fesovit was treated with the oxidizing mixture before following the rest of the procedure. This treatment changes ferric ion into ferrous ion. The column was filled with exchange material, and then loaded with sample. They were leached out with 1 M NH$_4$NO$_3$ as eluent and effluent was analyzed for the presence of Fe (III) by ethyle-diamine-tetra acetic acid (EDTA) titration using Cu-Pan as indicator (Varshney et al., 1994 a). The experiment was repeated 18 times (n = 18) for minimizing the error. The statistical analysis was made and the results are represented as the mean of 18 observations ± standard deviation (S. D.).

**Results and Discussion**

The average volumes of EDTA consumed in the titration of various effluent samples of SFH, SFR, SFD and FS were 9.07 ml, 7.40 ml, 2.87 ml and 9.07 ml, respectively. The average total iron calculated in these formulations was 96.11 ± 3.5 mg, 78.44 ± 1.5 mg, 30.37 ± 1.8 mg per 10 ml of each syrup, respectively and 47.7 ± 0.9 mg per spansole (Table 1 and Fig. 1).

Due to the absence of any standard regulations for the preparation of iron containing drugs of traditional system of medicine in India the formulations do vary in the quantity of constituents. That is quite evident from the observations of our study. Contrary to that the products of the modern medicine are prepared under strict regulations and good manufacturing practice is adopted.

![Figure 1 — Average total iron estimated in market samples of Sharbat-e-Faulad and Fesovit spansules (compared with their claimed/labeled composition)](image)

**Table 1 : Amount of total iron in the market samples of Sharbat-e-Faulad and Fesovit Spansules**

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Sample</th>
<th>Iron found in 10ml * Syrup &amp; each Spansole</th>
<th>S.D.</th>
<th>Iron claimed in 10ml Syrup &amp; each Spansole</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td><em>Sharbat-e-Faulad</em> (SFH) Hamdard (W) Lab., Delhi (Batch No. 60)</td>
<td>96.11 ± 3.5</td>
<td>72</td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td><em>Sharbat-e-Faulad</em> (SFR) Rex (U&amp;A) Remedies, Delhi (Batch No. 63/18)</td>
<td>78.44 ± 1.5</td>
<td>80</td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td><em>Sharbat-e-Faulad</em> (SFD) Dawakhana Tib. Col., A.M.U Aligarh. (Batch No. 35.36)</td>
<td>30.37 ± 1.8</td>
<td>120</td>
<td></td>
</tr>
<tr>
<td>4.</td>
<td>Fesovit Spansules (FS) Glaxo-Smith Kline, Bangalore (Batch No. 2006)</td>
<td>47.7 ± 0.9</td>
<td>150</td>
<td>(Dried FeSO$_4$ equal to about 45 mg elemental iron)</td>
</tr>
</tbody>
</table>

* Average of 18 observations
as mandatory. Therefore, chances of variation in the constituents are nonexistent. Fesovit spansule was found to contain the quantity of iron as specified by the manufacturer.

The iron content in Fesovit spansule was estimated and found to contain 47.7 ± 0.9 mg iron per spansule. It was almost equal to the content of iron supposed to be present in it as dried ferrous sulphate (FeSO₄·2H₂O) provides approximately 30% iron (Gilman et al, 1992; Laurence et al, 1997). The correctness of estimation of elemental iron in the spansule proved that this experimental design was highly precise.

The study revealed that the total iron content in SFH sample was 96.11 ± 3.5 mg/10 ml that is higher than the total iron content claimed (72 mg/10ml). If this drug is administered as recommended by the manufacturer (10ml twice daily) the content of iron provided by this formulation will be in the range of maximum content of iron given for the treatment of iron deficiency anemia (120-180 mg/day). The total iron content found in SFR sample was 78.44 ± 1.5 mg/10 ml which is almost equal to the iron content in this formulation as claimed by the manufacturer i.e., 80 mg/10 ml. If the syrup is administered in the dosage as recommended by the manufacturer, the amount of iron that will be available to the patient falls a little shorter of the maximum dose range for iron deficiency anemia. Still the amount of iron will be higher than that supplied by Fesovit spansule when administered as one spansule thrice daily. The total iron content found in SFD sample was estimated as 30.37 ± 1.8 mg/10ml, which is considerably lesser than the content of iron claimed by the proprietor i.e., 120 mg/10 ml. The content of iron provided by this formulation when administered in the recommended dosage (20 ml twice daily) is remarkably lower than the daily dosage for iron deficiency anemia. It is further resolved that the method is suitable for estimation of iron in the syrup as well as solid formulations of iron.

**Conclusion**

*Sharbat-e-faulad* manufactured by Hamdard (Wakf) Laboratories, New Delhi (SFH), contains the highest content of iron and among all the market samples is the most suitable for correcting iron deficiency anemia of any severity. *Sharbat-e-faulad* from Rex (U & A) Remedies (Pvt.) Ltd., New Delhi (SFR) appears to be a standard formulation because of the negligible discrepancy in the contents of iron claimed by the manufacturer and found after estimation. This formulation will fulfill the iron requirement to a considerable extent and therefore it may be relied upon for the treatment of iron deficiency anemia. *Sharbat-e-faulad* obtained from Dawakhana Tibbia College, AMU, Aligarh (SFD), has not been found a standard formulation because of the wide discrepancy between the claimed and estimated iron contents in this formulation. Moreover, the amount of iron provided by the recommended dosage is far less than the amount required for iron deficiency anemia. The method is suitable for estimation of iron in the syrup, as well as solid formulations of iron of herbo-mineral preparations of traditional system of medicine.

**References**


