




# Mössbauer investigation of $\text{FeCl}_3$ sediment after model water treatment by different aluminosilicate reagents

V. P. Filippov<sup>1</sup>  · V. A. Salomasov<sup>1</sup> · I. M. Kurchatov<sup>1</sup> ·  
N. I. Laguntsov<sup>1</sup> · D. Yu. Feklistov<sup>2</sup> ·  
R. A. Alexandrov<sup>1,2</sup> · Yu. V. Permyakov<sup>3</sup>

© Springer International Publishing AG 2017

**Abstract** The method of Mössbauer spectroscopy was used to study precipitated (coagulated) contaminations of model water with  $\text{FeCl}_3$  using reagents. Two different aluminosilicate reagents such as that produced by Ltd. “Trivektr” and a hybrid aluminosilicic reagent produced by JSC “Aquaservice” were used. The chemical state of iron in sediments was determined.

**Keywords** Mössbauer spectroscopy · Water treatment · Aluminosilicate reagents

## 1 Introduction

The ever-mounting deficit in clean drinking water for the growing world population requires the search for effective means of water purification, especially after its use at enterprises and in large cities. Artesian water resources in some regions are also contaminated. For example, underground artesian water resources in the Moscow region have an elevated dissolved iron content. The range of iron concentrations in this water varies from 0.5 to 20 mg/l, with a maximum permissible concentration of 0.3 mg/l. Hence, these water resources require purification before they can safely use as drinking water. To date, there are various methods

---

This article is part of the Topical Collection on *Proceedings of the 3rd Mediterranean Conference on the Applications of the Mössbauer Effect (MECAME 2017), Jerusalem, Israel, 5–7 June 2017*  
Edited by Mira Ristic, Stjepko Krehula, Israel Nowik and Israel Felner

---

✉ V. P. Filippov  
vpfilippov@mephi.ru

<sup>1</sup> National Research Nuclear University MEPhI (Moscow Engineering Physics Institute),  
31, Kashirskoe shosse, 115409, Moscow, Russia

<sup>2</sup> JSC Aquaservice, Kashirskoe shosse 31, 115409, Moscow, Russia

<sup>3</sup> Russian Metrological Institute of Technical Physics and Radio Engineering,  
141570, Mendeleevo, Solnechnogorskiy District, Moscow Region, Russia

of water purification, including the chemical method of cleaning with the help of coagulants and flocculants [1] and the method of using iron ferrates [2]. This paper is devoted to studying of influence of two reagents on iron concentration in model water and iron states in sediments. To purify of the model water with  $\text{FeCl}_3$  (concentration  $\sim 18$  mg/l) from impurities, an aluminosilicate reagent by Ltd. "Trivektr" and a hybrid aluminosilic reagent were used [3].

## 2 Experiment

### 2.1 Sample preparation and Mössbauer technique

The hybrid aluminosilic reagent was prepared in liquid state according to the procedure described in [3]. The aluminosilicate reagent "Trivektr" was added in a similar dosage in terms of  $\text{Al}_2\text{O}_3$  to the aqueous  $\text{FeCl}_3$  solution. A dose of 1 mg/l of a 100% solution of this reagent in terms of  $\text{Al}_2\text{O}_3$  was added to the same model water. After the addition of the aluminosilicate reagent "Trivektr" the processes of coagulation and flocculation took place. But after the addition of the hybrid aluminosilic reagent to the solution, the processes of coagulation, flocculation and sorption started to occur. In the second case, after adding hybrid aluminosilic reagent, the chemical cleaning process occurred along with an additional sorption processes, in which pollutants formed into matrix-like microporous and mesoporous zeolite structures. Sediments precipitated from solution more rapidly after aluminosilic reagent.

The sediments of the two model waters were air dried at  $t = 60$  °C. Mössbauer absorbers (samples 1 and 2) were prepared from the dried sediments by mixing with paraffin. Mössbauer spectra were obtained in the transmission mode at room temperature. The detector was a thin NaJ (Tl) crystal (200  $\mu\text{m}$ ). The spectrometer was calibrated using a standard absorber, a thin foil of  $\alpha$ -Fe. The source of gamma radiation was the  $^{57}\text{Co}$  isotope in the Cr matrix. The spectra were fitted using Univem MS program. The relative content of the phases were determined from the assumption that the resonance absorption probabilities for all phases are the equal.

## 3 Result and analysis

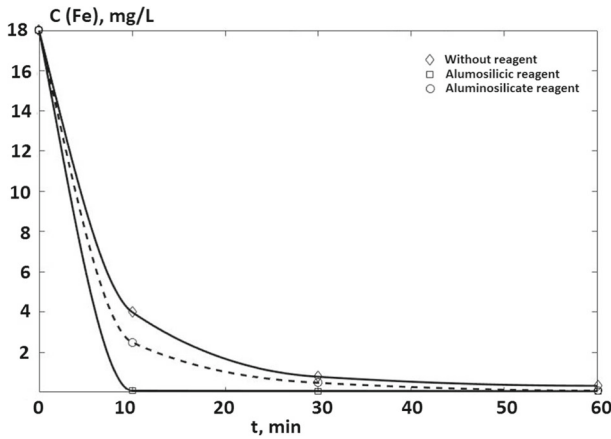
### 3.1 Chemical analysis

Figure 1 shows the kinetics of clarification of model waters after treatment by two reagents. The graph shows the variation of suspended substance (iron ions) versus time.

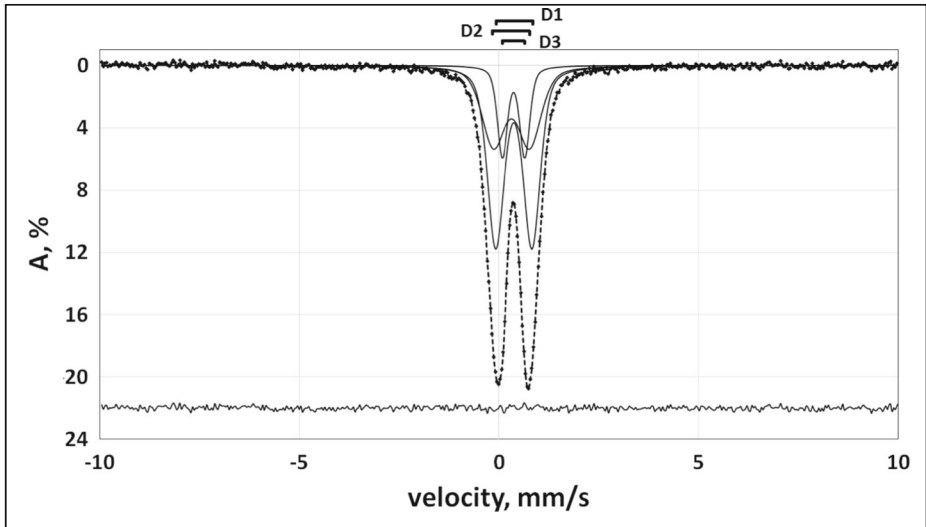
Analysis of kinetic curves (Fig. 1) shows that the hybrid aluminosilic reagent reduces the iron concentration to  $C < 0.01$  mg/l within  $t = 10$  min. Meanwhile, during the same time interval  $t = 10$  min, the aluminosilicate reagent "Trivektr" reduces the iron concentration to only  $C \sim 2.4$  mg/l. This result indicates that the hybrid aluminosilicate reagent acts more efficiently than "Trivektr". The two sediments formed by the action of the reagent were used to analyze the iron state by the method of Mössbauer spectroscopy.

### 3.2 Mössbauer investigation

Figure 2 shows the Mössbauer spectrum of  $^{57}\text{Fe}$  in sediment obtained from the model water with  $\text{FeCl}_3$  after treatment by the aluminosilicate reagent "Trivektr".



**Fig. 1** The iron concentration variation in water after treatment by various reagents

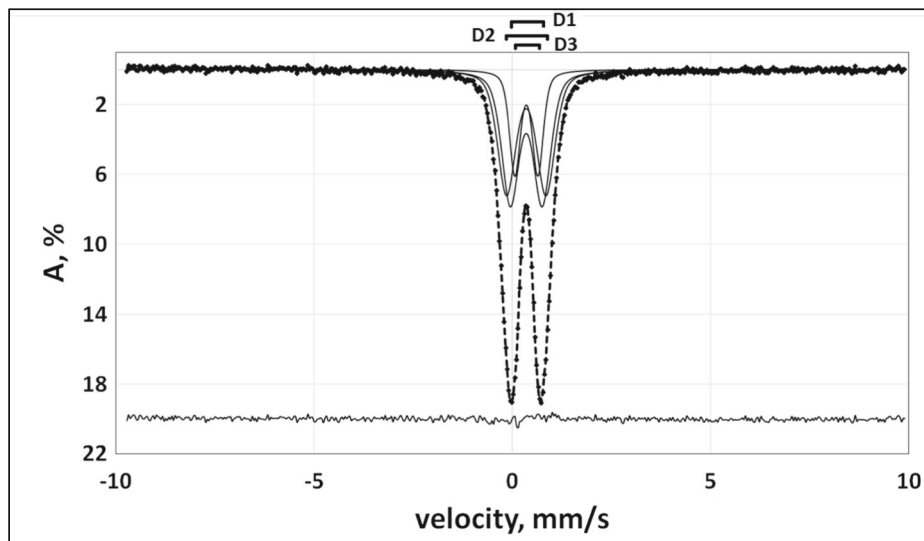


**Fig. 2** Mössbauer spectrum of  $^{57}\text{Fe}$  of the dried sediment obtained after treatment model water containing  $\text{FeCl}_3$  using aluminosilicate reagent “Trivektr”

The Mössbauer spectrum is a superposition of three lines of hyperfine quadrupole splitting of paramagnetic phases. The parameters of the spectral lines obtained are presented in Table 1. The most intensive paramagnetic phase D1 with spectral parameters  $IS = 0.34 \pm 0.05 \text{ mm/s}$  and  $QS = 0.82 \pm 0.05 \text{ mm/s}$  is interpreted as a trivalent iron compound, the relative content of which is 44% of the total concentration of iron. The paramagnetic phase D2 with spectral parameters  $IS = 0.35 \pm 0.05 \text{ mm/s}$  and  $QS = 1.06 \pm 0.05 \text{ mm/s}$  can be interpreted as a ferric compound with atomic positions in a more elongated unit cell. This conclusion was made on the basis of ref. [4]. The paramagnetic phase D3 with spectral parameters  $IS = 0.35 \pm 0.05 \text{ mm/s}$  and  $QS = 0.58 \pm 0.05 \text{ mm/s}$  can be interpreted as a ferric compound with a relative concentration of 22%. Since the model solution contains

**Table 1** Mössbauer parameters of sediment obtained from contamination of model water with FeCl<sub>3</sub> after treatment by the aluminosilicate reagent “Trivektr”

Phase	IS, mm/s	QS, mm/s	S <sub>rel</sub> , %	G, mm/s
Doublet 1	0.34 ± 0.05	0.82 ± 0.05	44	0.52
Doublet 2	0.35 ± 0.05	1.06 ± 0.05	34	0.53
Doublet 3	0.35 ± 0.05	0.58 ± 0.05	22	0.33

**Fig. 3** Mössbauer spectrum of <sup>57</sup>Fe of the dried sediment obtained after treatment model water containing FeCl<sub>3</sub> using hybrid aluminosilicic reagent

Cl<sup>-</sup> ions, all three paramagnetic phases can be interpreted as the compound  $\beta$ -FeOOH in which iron atoms are in trivalent states [4, 5]. The authors of refs. [4, 5] have proposed that the room temperature spectrum must be fitted with three quadrupole doublets, where the intensity of two of them is always in the 2:1 ratio. In Table 1 one can see this ratio between doublets 1 and 3. Two doublets differ by the relative orientation of the principal axis of the electric field gradient with respect to the fourfold axis of symmetry. They argue that the replacement of Cl<sup>-</sup> ions by OH<sup>-</sup> ions in the structure is an important factor for the presence of different iron sites.

Figure 3 shows the Mössbauer spectrum of <sup>57</sup>Fe in sediment obtained from the model water with FeCl<sub>3</sub> after treatment by the hybrid aluminosilicic reagent. The Mössbauer spectrum is a superposition of three lines of hyperfine quadrupole splitting of paramagnetic phases. The external types of spectra in Figs. 2 and 3 under the influence of different reagents have a similar shape. The obtained parameters of the spectral lines are presented in Table 2.

The paramagnetic phase D1 is described by a doublet with spectral parameters IS = 0.36 ± 0.05 mm/s and QS = 0.89 ± 0.05 mm/s is interpreted as a ferric trivalent compound. In contrast to the spectrum of Fig. 1, this phase contains 51% of the total iron, which is higher relative to the iron content precipitated by the reagent “Trivektr”. The paramagnetic phase D2 with spectral parameters IS = 0.32 ± 0.05 mm/s and QS = 0.91 ± 0.05 mm/s can also be interpreted as a trivalent iron compound with atomic positions in a more elongated unit

**Table 2** Mössbauer parameters of sediment obtained by treatment of model water by hybrid aluminosilicic reagent

Phase	IS, mm/s	QS, mm/s	S <sub>rel</sub> , %	G, mm/s
Doublet 1	0.36 ± 0.05	0.89 ± 0.05	51	0.52
Doublet 2	0.32 ± 0.05	0.91 ± 0.05	35	0.53
Doublet 3	0.36 ± 0.05	0.55 ± 0.05	14	0.49

cell. The content of this phase in the sample is equal to the content of phase D2 in the first sample. The paramagnetic phase D3 with spectral parameters IS = 0.36 ± 0.05 mm/s and QS = 0.55 ± 0.05 mm/s can be interpreted as a trivalent iron compound. The phase D3 has a concentration of 14% relatively to the total iron content. Taking into account the fact that the model solution contains Cl<sup>1-</sup> ions, all three paramagnetic phases in second specimen can also be characterized as the compound β-FeOOH [4].

The analyses show that hybrid aluminosilicic reagent is a more efficient reagent for reducing the iron content in water. This is clearly seen in Fig. 1: the bulk of the dissolved iron in the water has precipitated within 10 min. In addition, the residual concentration of iron in water is less than when treated with another reagent. Mössbauer studies showed that when processing with a more efficient reagent (hybrid aluminosilicic reagent), small changes occurred in the parameters of the Mössbauer spectra, such as IS and QS of the second phase. IS within the margin of error decreased, and QS decreased from 1.06 ± 0.05 to 0.91 ± 0.05 mm/s. The most noticeable changes occurred in relative concentrations of phases 1 and 3. The concentration of phase 1 increased from 44% to 51%, while the concentration of phase 3 decreased. It can be assumed that the higher efficiency of the hybrid aluminosilicic reagent is interrelated with changes in the parameters of the Mössbauer spectra.

## 4 Conclusions

Experiments have been performed to purify contaminants in model water with FeCl<sub>3</sub> using the hybrid aluminosilicic reagent produced by the JSC “Aquaservice” and using the aluminosilicate reagent of the Ltd. “Trivektr”. Based on the analysis of Mössbauer spectra and chemical analysis, the following conclusions are drawn:

- 1) The hybrid aluminosilicic reagent is more effective than “Trivektr” reagent. With the prior reagent, the iron concentration decreased to 0.01 mg/l (Fig. 1), whereas with the latter reagent, the iron concentration decreased only to ~2.4 mg/l (Fig. 1—dotted line).
- 2) It has been suggested that the hybrid aluminosilicic reagent exhibits high efficiency due to existence of the sorption mechanism, as evidenced by the formation of matrix-like microporous and mesoporous zeolite structures. The indicator of the efficiency of the hybrid aluminosilicic reagent can be the fact that the relative iron content of the phase 1 (Table 2, D1 with IS = 0.36 ± 0.05 mm/s and QS = 0.89 ± 0.05 mm/s) was increased by ~16%, whereas the iron content of other phase 1 (Table 1 D1 with IS = 0.34 ± 0.05 mm/s and QS = 0.82 ± 0.05 mm/s) decreased.
- 3) The formed chemical compound of iron in the sediment was identified as β-FeOOH. Relative concentrations of different type of β-FeOOH depend on the reagent.

**Acknowledgements** The work was financially supported by MEFPI’s Competitiveness Program and the Ministry of Education and Science, agreement No 14.575.21.0086. Unique project identifier RFMEFI57514X0086.

## References

1. Kurchatov, I.M., Laguntsov, N.I., et al.: The reagent-sorption technology of water treatment. *J. Phys. Procedia* **72**, 89–92 (2015)
2. Dedushenko, S.K., Perfiliev, Yu.D., Kulikov, L.A.: Mössbauer spectroscopy and quality control in ferrate technology. *Hyperfine Interact.* **218**, 59–65 (2013)
3. Filippov, V.P., Kurchatov, I.M., Laguntsov, N.I., et al.: Mössbauer investigation of a nepheline substrate used for preparing a water treatment reagent. *Hyperfine Interact.* **237**, 136–140 (2016)
4. Alenkina, I.V., Oshtrakh, M.I., Klencsar, Z., et al.:  $^{57}\text{Fe}$  Mössbauer spect. and electron param. resonance studies of human liver ferritin, Ferrum Lek and Maltofer. *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **130**, 24–36 (2014)
5. Rézel, D., Génin, J.M.R.: The substitution of chloride ions to  $\text{OH}^-$  -Ions in the akaganeite beta ferric oxyhydroxide studied by Mössbauer effect. *Hyperfine Interact.* **57**, 2067–2076 (1990)