

Elution Evaluation of Silver Compound-containing Antibacterial Fabric by Atomic Absorption Spectrophotometer

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User Benefits

- ◆ Analysis of silver in the eluate of antibacterial fabric is possible by atomic absorption spectrophotometer (AA).
- ◆ By using both flame and furnace method, efficient measurement from high concentration to low concentration is feasible.

Introduction

With the coronavirus (COVID-19) epidemic, silver ions (Ag^+) are drawing attention again. Because of their high antibacterial properties, harmlessness to humans, and long-lasting antibacterial effects, silver compounds are widely used as inorganic antibacterial agents, and many daily necessities with added silver are commercially available. Previous studies have confirmed that Ag^+ has antibacterial effects against the influenza virus, common coronavirus, MRSA, etc.

Herein, we introduce an example of evaluating the elution volume of silver (Ag) from an antibacterial fabric containing silver compound using the atomic absorption method. By using both the flame method, which enables rapid measurement, and the highly sensitive furnace method, it is possible to analyze eluted Ag in a wide concentration range.

Sample & Elution Evaluation

A commercially available Ag^+ -containing antibacterial fabric was used as the measurement sample. Two sample pieces were cut out from one piece of fabric (about $5\text{ cm} \times 5\text{ cm}$), one was used for the elution test, and the other was used for the decomposition. The amount of Ag in the decomposed solution was considered the total amount of Ag in the antibacterial fabric, and the elution rate from the fabric was calculated. Fig. 1 shows the decomposition process flow.

The elution test was performed with the procedure shown in Fig. 2, and the obtained solution was used as the eluate for 1 day. After drying the sample piece, the elution test was performed for 15 days by repeating the steps in Fig. 2.

- Put the sample piece ($5\text{ cm} \times 5\text{ cm}$, 0.68 g) in a beaker
↓ ←Add small amount of water and 5 mL of nitric acid
- Heat at about $100\text{ }^\circ\text{C}$ on the hot plate covered with a watch glass
↓
- After the violent reaction has subsided, raise to $190\text{ }^\circ\text{C}$ and decompose by heat
↓
- Heat decomposition until the amount of liquid decreases and it begins to turn brown
↓
- Remove from hot plate
↓ ←Add a small amount of nitric acid
- Repeat steps 4 and 5 and add nitric acid until it becomes a pale yellow color
↓
- Concentrate until almost dry
↓ ←Add about 0.1 mL of concentrated nitric acid
- Cool down
↓
- Filtration
↓
- Increase volume to 20 mL with pure water, then dilute 3 times with water

Fig. 1 Decomposition Process of Antibacterial Fabric

- Put the sample piece ($5\text{ cm} \times 5\text{ cm}$, 0.65 g) in 25 mL of pure water and leave it for 24 hours
↓
- Place in ultrasonic cleaner for 10 minutes
↓
- Squeeze the sample piece and collect the eluate
↓
- Add small amount of nitric acid to ③, and use it as measurement solution

Fig. 2 Elution Test of Antibacterial Fabric

Instrument Configuration and Measurement Conditions

The instrument used here was a Shimadzu AA-7000F/AAC atomic absorption spectrophotometer with a graphite furnace atomizer (automatic switching between the flame and furnace methods is possible) and an autosampler.

Table 1 shows the measurement conditions. The measurement was carried out by the calibration curve method.

Table 1 Measurement Conditions

Element		Ag
Analysis wavelength		328.1 nm
Slit width		0.7 nm
Lighting mode		BGC-D2
Lamp current		10 mA
Flame Method	Flame type	Air- C_2H_2
	C_2H_2 flow rate	2.2 L/min
	Integration time	3 s
	Repetition	3 times
Furnace Method	Ashing temp.	$400\text{ }^\circ\text{C}$
	Atomization temp.	$1800\text{ }^\circ\text{C}$
	Tube type	Platform tube
	Injection volume	Sample $10\text{ }\mu\text{L}$ + Pd (50ppm) $2\text{ }\mu\text{L}$
	Signal processing	Peak height
	Repetition	2 times (max 3 times)

Measurement Results

The decomposition solution and the eluate for the first 10 days were measured by the flame method first.

The calibration curve of the flame method is shown in Fig. 3.

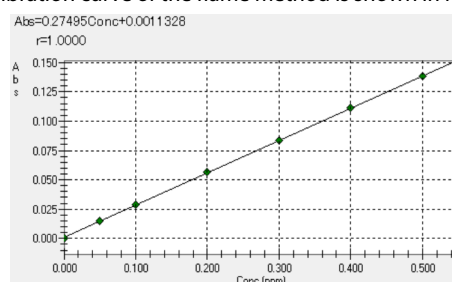


Fig. 3 Calibration Curve of Ag by Flame Method

From the 7th day of immersion of the antibacterial fabric, the Ag concentration in the eluate became lower than the limit of quantification (LOQ) by the flame method. Therefore, after the 7th day, measurement was performed by the highly sensitive furnace method. The calibration curve of the furnace method is shown in Fig. 4.

Table 2 shows the measurement results of the antibacterial fabric. The concentration of eluate is the value converted to the concentration in the solution before dilution, and the actual concentration is the value obtained by converting the measured concentration to the concentration in the solid.

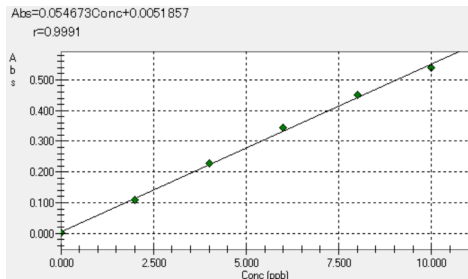


Fig. 4 Calibration Curve of Ag by Furnace Method

Fig. 5 shows the Ag concentration and elution rate changes in the eluate over 15 days. The elution rate was calculated by dividing the integrated value of the Ag concentration in the elution by the Ag concentration in the decomposed solution (58 µg/g).

A large amount of Ag was eluted during the first 5 days, but this decreased gradually, and after 9 days later, the elution amount tended to be almost unchanged.

Conclusion

The AA-7000 series has a lineup of a dual atomizer system and dedicated machines with the rapid flame method or highly sensitive furnace method.

With the flame method, it is possible to measure the total Ag concentration easily in the antibacterial fabric and the change in elution amount for up to about 1 week in the elution test. If it is necessary to measure the lower concentration range, such as when evaluating the period until the Ag elution from the antibacterial fabric disappears, it can be analyzed by the highly sensitive furnace method. The dual atomizer system or a dedicated machine could be selected depending on the purpose.

Table 2 The Measurement Results of Antibacterial Fabric

F R A M E	Days	Fabric	D1	D2	D3	D4	D5	D6	D7	D8
	Conc. of Eluate (mg/L)	0.26	0.064	0.081	0.13	0.072	0.086	0.048	<0.03*	<0.03*
	SD	1.1	3.5	0.78	0.63	0.77	0.57	1.9	0.78	0.45
	%RSD	0.44	5.5	0.97	0.48	1.1	0.66	4.0	3.0	2.4
Actual Conc. (µg/g)	58	2.2	3.1	5.1	2.7	3.3	1.8	<1.2*	<1.2*	
F U R N A C E	Days	D7	D8	D9	D10	D11	D12	D13	D14	D15
	Dilution Rate	5	5	2	2	2	2.5	2	2	2
	Measurement Conc. (µg/L)	5.5	4.8	4.9	4.9	6.2	7.8	7.2	5.8	4.4
	Conc. of Eluate (mg/L)	0.027	0.024	0.0099	0.0098	0.012	0.020	0.014	0.012	0.0089
	SD	0.0063	0.024	0.0022	0.0003	0.0003	0.0018	0.0021	0.0073	0.0042
	%RSD	0.61	2.56	0.58	0.07	0.07	0.24	0.38	1.63	1.23
	Actual Conc. (µg/g)	1.0	0.92	0.38	0.37	0.47	0.75	0.55	0.45	0.34

* Indicates LOQ or lower. (LOQ was calculated from the calibration curve at a concentration of 0.01 Abs.)

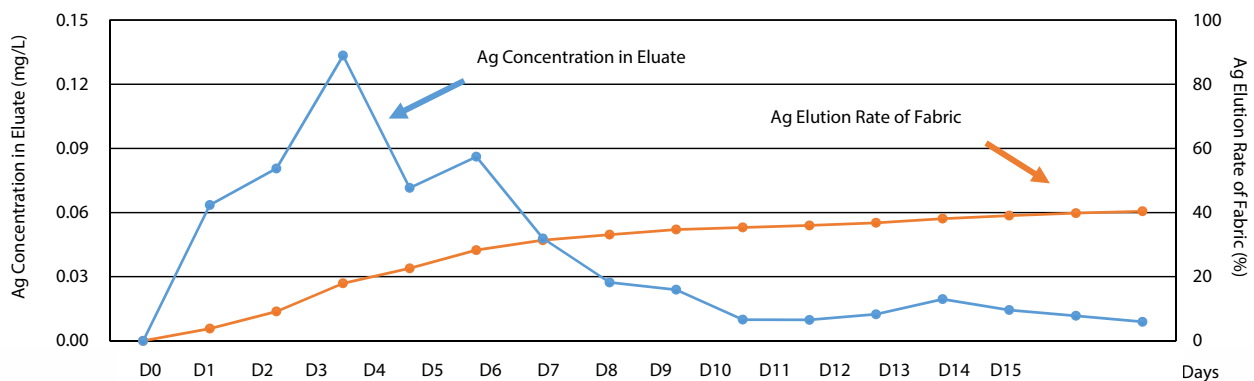


Fig. 5 Ag Concentration and Elution Rate Changes



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