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Short communication

Determination of tripolyphosphate in frozen cod and scallop adductor by ion chromatography

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Abstract

We introduce a method for the determination of tripolyphosphate in frozen cod and scallop adductor by using ion chromatography. The tripolyphosphate was extracted from minced cod and scallop adductor with deionized water by ultrasonic leaching, and then the proteins soluble in water were precipitated with trichloroacetic acid and removed by filtering. An ion chromatograph with an ionpac AS11-HC anion-exchange column, an ASRS (Anion Self Regenerating Suppression), a conductivity detector and a gradient pump (sodium hydroxide gradient) was used. The detection limit was below 5 mg tripolyphosphate/kg cod or scallop adductor. This method is applicable to the determination of polyphosphates in aquatic products and the procedures are easy to implement.

Keywords: Fish; Food analysis; Tripolyphosphate

1. Introduction

Sodium tripolyphosphate is used as a quality-improving agent in the aquatic product process; but it is forbidden to use it in some aquatic processes, such as cod and scallop adductor processes. It is difficult to judge whether tripolyphosphate is used in the processing of frozen cod and scallop adductor using conventional analytical methods [1,2] because of soluble phosphate contained in cod and scallop adductor. Ion chromatography has been used in the separation of polyphosphates [3–5] and characterizes polyphosphates chain length [5]. This paper introduces a method of determining tripolyphosphate in frozen cod and scallop adductor. The tripolyphosphate in frozen cod and scallop adductor was extracted with deionized water and determined using an ion chromatography system.

2. Experimental

2.1. Ion chromatography instrument

The instrument was a Dionex Model 500 ion chromatograph (Sunnyvale, CA, USA) with a GP 40 gradient pump, an ED 40 electrochemical detector, a Dionex IonPac AG 11-HC (50 mm×4 mm) and IonPac AS11-HC (250 mm×4 mm) columns, a 25 μl sample loop and an Anion Self-Regenerating suppressor (ASRS) which was operated in the auto-suppression recycle mode. The suppressor current setting was 300 mA and the detector full scale setting was 2666 μs. Data acquisition and instrument control was performed using the Dionex Peaknet 5.01 program.

2.2. Reagents

Deionized distilled water with a specific resistance

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of >18.2 MΩ from a Millipore (Bedford, MA, USA) Milli-Q plus PF water purification system. This deionized water was used to prepare all reagents and standards.

Fifty percent (w/w) NaOH was prepared using sodium hydroxide pellets (Guaranteed Reagent, Merck). One hundred millimolar and 25 m*M* NaOH were made by dilution from 50% NaOH with degassed deionized water.

Trichloroacetic acid (20%) was prepared by dissolving 200 g trichloroacetic acid in 800 ml deionized water.

The stock tripolyphosphate solution (1 mg/ml) was prepared by dissolving 151.5 mg $Na_3P_3O_{10}$ (Guaranteed Reagent, 96%) in deionized water and diluting to 10 ml.

2.3. Sample preparation

A sample of frozen cod was minced prior to analyses. A 10-g amount of minced cod was collected in a 200-ml beaker, and 50 ml deionized water was added. After waiting for about 10 min until there was no ice, the solution was extracted for 10 min by ultrasonic leaching. Fish meat was removed by filtering (filter paper No. 42, Whatman), then 5 ml trichloroacetic acid was added to the solution, and the precipitate was removed by filtering. The solution was adjusted to pH>8 with 2 mol/l sodium hydroxide and was diluted to 100 ml.

The sample solution of scallop adductor preparation was the same as frozen cod.

2.4. Operating procedure

A sodium hydroxide gradient was used (Table1). Eluent flow-rate was 1 ml/min. The sample solution was diluted tenfold, and was injected through a 0.45 μm filter before entering the chromatography system.

Table 1
Sodium hydroxide gradient condition

Time	%E1=25 m*M*	%E2=100 m*M*
Init	100	0.0
0.0	100	0.0
5.0	100	0.0
15.0	0.0	100
18.0	0.0	100
20.0	100	0.0
25.0	100	0.0

3. Results and discussion

Sodium tripolyphosphate is soluble in water so it can be extracted with deionized water and mincing making extracting easy. Experiments showed (Fig. 1) that 5 min ultrasonic leaching can reach extraction equilibrium. Proteins soluble in water may contaminate anion-exchange columns and had to be precipitated by trichloroacetic acid and removed by filtering. Polyphosphates are not stable in aqueous solution; high temperature and low pH will accelerate hydrolysis of polyphosphates. The sample solution was adjusted to pH>8 with sodium hydroxide, and the sample should be analyzed within 2 h. It was verified that tripolyphosphate in 0.1% choloracetic acid had not obvious change within 10 h by our experiments.

Ion chromatography analysis of tripolyphosphate in cod and scallop adductor is shown respectively in Figs. 2 and 3. Tripolyphosphate can well be separated from the matrix by the suggested method, tricholoracetic acid (TCA) is eluted last.

Linearity was investigated using the stock solution which was diluted serially. Response of tripolyphosphate was linear in the working range from 1 to 100 mg/l, the equation was $y=2.260\cdot10^{-5}x$ and the correlation coefficient r^2 for a linear least square fit was 0.9991. Nine duplicates were tested and the RSD was 3.65%. The detection limit was 5 mg/kg which means that tripolyphosphate contents >5 mg/kg in cod or scallop adductor can be detected.

An anion-exchange column AS11-HC was used in this paper. Because of its high capacity, the retention time of tripolyphosphate is longer than that of AS11.

The results obtained by the proposed method using samples of cod and scallop adductor were shown in Table 2.

4. Conclusion

Tripolyphosphate in cod and scallop adductor was extracted in water and was separated and determined

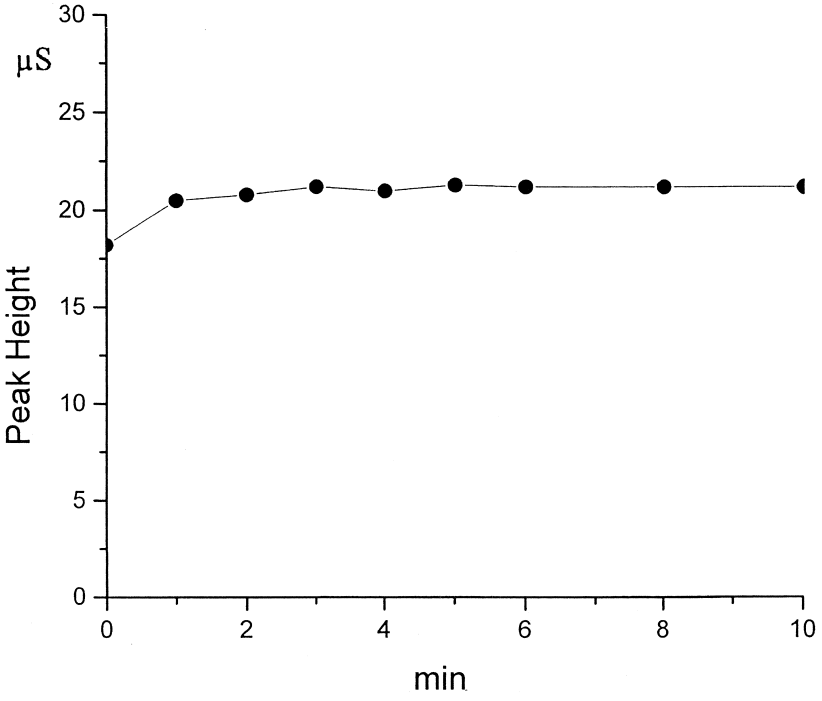

Fig. 1. Ultrasonic leaching curve.

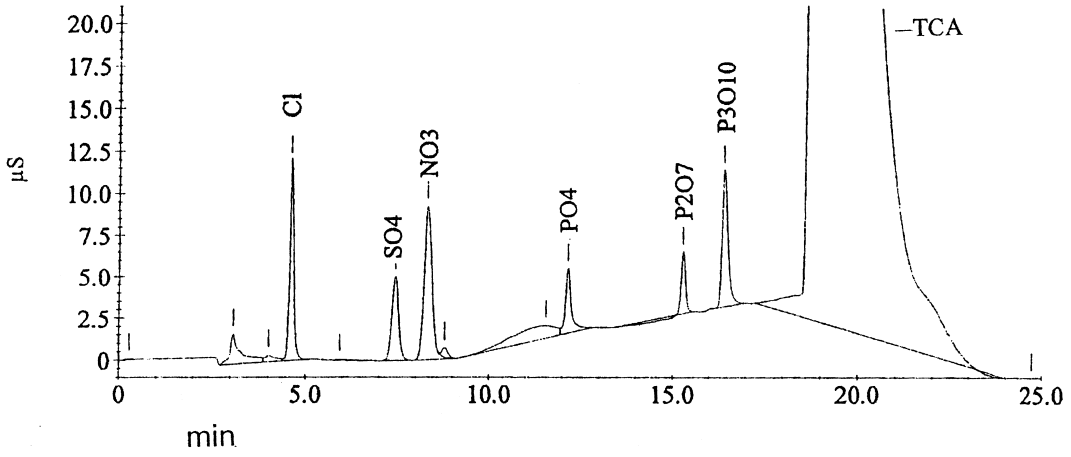

Fig. 2. Chromatogram of tripolyphosphate in sample of cod. Retention times: Cl=4.62, SO_4=7.50, NO_3=8.28, P_2O_7=15.10, P_3O_{10}=16.15, TCA=18.65 min.

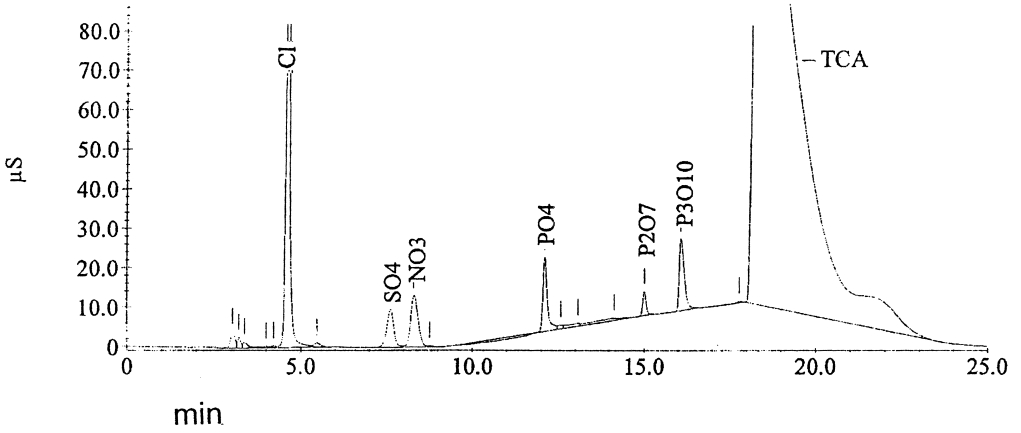

Fig. 3. Chromatogram of tripolyphosphate in sample of scallop adductor. Retention times: Cl=4.60, SO_4=7.42, NO_3=8.30, PO_4=12.13, P_2O_7=15.21, P_3O_{10}=16.30, TCA=18.82 min.

Table 2
Tripolyphosphate content in samples of cod and scallop adductor

Sample	Content (mg/g)	Added[b] (mg)	Recovery (%)
Cod 1#	0.85	0.4	88.6
		2.0	100.2
		4.0	95.1
Cod 2#	0.0092	0.4	92.5
		2.0	98.8
		4.0	90.6
Scallop adductor 1#	3.8	0.4	93.4
		2.0	89.7
		4.0	104.9
Scallop adductor 2#	ND[a]	0.4	89.2
		2.0	91.3
		4.0	96.7

[a] Not detected, tripolyphosphate content in sample is below detection limit.
[b] Tripolyphosphate added in per g sample.

using ion chromatography. The procedures were easy to implement.

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