Iron in Photographic Gelatin — II
Effective Elimination of Iron Impurity in Gelatin

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Abstract

Comparing some previous ion exchange methods, ion exchange resin with larger pores structure is better than gel ion exchange resin for elimination iron impurity in gelatin. But it is possible to dramatically increase the efficiency to remove iron by means of changing chemical form of iron as a result of changing in the iron valence even with gel resin. Consequently, a novel method to eliminate iron impunty for prepartion of photographic gelatin is developed.

Preface

The experimental results demonstrated that the iron impurity in photographic gelatin remarkably damage photographic performances of silver halide emulsion.[1] Therefore, it is necessary to remove the iron impurity in gelatin as far as possible so as to be utilized in photographic film making. It is suggested that the amount of iron in photographic gelatin with high quality should be less than 2ug/g,[2] An effective way to remove iron and other impurities during making of photographic gelatin is ion exchange technique. Moreover much effort has been made in order to improve the effficency of removal of gelatin iron. For example, a small amount of calcium salt or a sodim salt is added to the gelatin solution prior to contact with ion exchange resin, resulting in fast movement of iron complex. Therefore, the removal of Fe from gelatin is increased.[3] In late 20 years, a new kind of ion exchange resin with large pores structure has been developed which posssesses larger and much more pores inside than that of common gel resin. These features are favoable to remove impurities from biomacromolecules because of increasing of exchange chance between impurities and functions groups. So it is also applied to purify gelatin.[4]

And yet the efficiency of ion exchange is not only influenced by the factors mentioned above but also connected with the size of iron-containing molecules. There are Fe^{2+} ions and Fe^{3+} ions in Gelatin,[5] Fe^{3+} is reduced by ascorbic acid to Fe^{2+} in gelatin and the Fe^{2+} ions produced are dcominantly bound to smaller molecules than Fe^{3+} ions do.[6] On this base, it is possible to increase the efficiency to remove iron by mcans of changing of chemical form of iron as a result of reducing of Fe^{3+} ions to Fe^{2+} ions. In this work, the related study is reported.

Materials and Methods

The gelatin sample for this experiment is listed in Table l.

Table 1. The Features of Gelatin Sample

gelatin sample	Fe^{2+} content (ug/g)	Fe^{3+} content (ug/g)	viscosity(cp) 6%soln.; 60°C
QPA	7.50	16.50	5.74

A strong acid cation ion exchange resin in the gel form and a cation ion exchange resin with large pores structure, which has amino-groups and phospho-groups. are used in this study First of all, these two kinds of resins should be cleaned carefully in the follwing way. respectively. After socking in water for 24 hours, the resin is treated with 50% ethyl solution, 5% HCl solution, distilled water and 5% NaOH solution successively. Then it is washed with distilled water till PH = 7 and subsequently soaked in the 0.6% Na_2EDTA solution for 24 hours. At last the Na_2EDTA solution is poured out and the resin is nnsed again with the distilled water.

The contents of Fe^{2+} ions and Fe^{3+} ions in gelatin solution are determined with spectrophotometic methods,[7] respectively.

Experimental Procedure

500ml of 5% QPA gelatin solution was passed through the cation exchange resin in the gel form and subsequently over an ion exchange resin with the flow rate of 4mlmin-1 at 60°C. The first 100ml of elute was rejected and other 400ml of elute was collected. Then its iron content was determined.

50g of ascorbic acid was added into another 500ml of 5% QPA gelatin solution nad this mixed solution stood for over 2 hours. After filtrating of residual ascorbic acid, the gelatin solution was treated in the same way as the above experimental procedure.

After replacing of the gel resin with the cation ion exchange resin vvith large pores structure, the third 500ml of 5% QPA gelatin solution without addition of ascorbic acid was circulated after the same fashion as the above experimental procedure.

In addition the contents of Fe^{2+} and Fe^{3+} in twenty gelatin samples were determined, respectively.

Results and Discussion

The efficiencies of removal of gelatin iron in various manners are shown in Table 2

The contents of Fe^{2+} and Fe^{3+} are listed in table 3 in some gelatin samples.

The results in Table 2 indicate that the efficiency of the ion exchange resin with large pores structure is indeed higher than that of gel resin for elimination of gelatin iron. It is surprising that this efficiency is powerfully increased even using the gel resin provided that the gelatin solution is treated by ascorbic acid prior to contact the gel resin. In such a manner, the gelatin iron content is dramatically reduced from 1.2 ug/ml to 0.06 ug/m1. But in the case of gelatin solution without addition of ascorbic acid, the iron content is merely reduced to 0.48 ug/ml. Therefore, combination of ascorbic acid treatment of gelatin solution with the ion exchange technique offers a novel method to eliminate effectively iron impurity in gelatin. In comparison with the resin with large pores structure, this method are more convenient and more economical.

It is known from Table 3 that the Fe^{3+} contents are generally higher than Fe^{2+} contents in the tested 20 gelatin samples, especially in the gelatin samples with higher total iron contents. It implies that the novel method developed in this paper has possibility of application.

Table 2. Elimination of Gelatin Iron

tested solution	resin	Fe content of soln. Before exchange (ug/ml)	Fe content of soln. After exchange (ug/ml)
5%QPA gelatin soln.	Gel resin	1.2	0.48
5%QPA gelatin soln. Pre treated by ascorbic acid	Gel resin	1.2	0.06
5%QPA gelatin soln.	Gel resin with large pores size resin	1.2	0.20

Table 3. Contents of Fe^{2+} and Fe^{3+} respectively in different gelatin samples(ug/g)

Samples	K3	CHB962	CHB981	FD	BP	G5S	G56	G57	CHB30	CHB70
Fe^{2+} cont.	4.53	2.90	2.90	2.30	3.40	2.80	2.80	2.80	3.80	3.60
Fe^{3+} cont.	0.00	3.30	3.10	1.30	2.70	1.90	1 90	19.80	22.30	
Total iron.	4.53	6.20	6.20	5.40	4.70	5.50	4.70	4.70	23.6	25.90
Samples	**CHB90**	**QPA4**	**QPA5**	**JX**	**F51784**	**F52354**	**G58**	**G59**	**GK**	**GJ**
Fe^{2+} cont.	2.50	1.60	4.00	3.50	1.00	2.00	2.80	3.40	5.40	2.50
Fe^{3+}cont.	21.40	23.30	17.90	7.90	8.90	6.70	3.10	2.50	2.40	5.00
Total iron.	23.90	24.90	21.90	11.40	9.90	8.70	5.90	5.90	7.80	7.80

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