# 17. Production of Fine Powder from silk by Radiation

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## Abstract

Silk fine power was prepared directly from silk fiber irradiated with an accelerated electron beam(EB). Irradiated silk fiber was well pulverized only by physical crushing using ball mill without any chemical pretreatment. Raw and degummed silk fibers were irradiated at ambient temperature in the dose range of 250 - 1000 kGy. Although unirradiated silk fibers were not pulverized at all, irradiated fibers were easily pulverized and showed higher conversion from fiber to powder for higher doses. The presence of oxygen in the irradiation atmosphere enhanced pulverization of silk fiber. Raw silk fibers were less pulverized compared to degummed ones. The electron microscope observation showed that the minimum particle size of silk powder obtained from fiber irradiated by 1000 kGy in oxygen was less than 10 microns. It was found that fibroin powder obtained in this work dissolved remarkably into cold water, thought unirradiated fibroin fiber had little solubility even in hot water. A typical soluble fraction was about 60% for fibroin powder obtained from fiber irradiated by 1000 kGy in oxygen.

**Key words :** Silk, Powder, Protein, Fibroin, Radiation degradation, Dissolution

## 1. Introduction

Silk powder is one of the useful physical forms of silk protein as well as fiber and film, for the application as biopolymer or biomaterial[1]. Silk powder has already found its utility as cosmetic materials and functional foods. It is, however, unexpectedly difficult to fabricate silk powder directly by mechanical processing on silk in the form of fiber because of its extremely high impact break energy. Thus, fine particles for cosmetic materials are produced from fibroin aqueous solutions. In such a typical conventional method, a concentrated neutral salt solution[2] is necessary to dissolve fibroin fiber with subsequent dialysis treatment, since silk fibroin does not dissolve in water. In this work we represent how the radiation degradation technique is effective for silk fine powder fabrication and also the unexpected finding that fibroin powder obtained in this work dissolves remarkably into cold water.

## 2. Experimental

### 2.1 Materials

Raw and degummed silk fibers from the commercial silk worm variety of *Bombyx Mori* were used in this work. An enzymatic treatment was adopted to remove the sericin, which surrounds the silk fibroin fiber. Each sample of about 1g was packed into a plastic bag, filled with nitrogen or oxygen gas and sealed by heating.

### 2.2 Irradiation

Irradiation was carried out at room temperature with 1 MeV electron beam. Samples to be irradiated were transferred under scanned electron beam of 1 mA at a speed of 1.17 m/min to yield a dose of 50 kGy/pass. Total doses ranged from 250 to 1000 kGy.

### 2.3 Pulverization

A ball mill, Frisch Pulverisette type 06, was used to pulverize irradiated fiber samples. An initial sample loading was about 0.8g. Before loading, a fiber sample was cut into

about 1 cm long. A milling time was fixed to 1 h. After milling, crushed samples were filtered with 90 micron stainless steel mesh. Because of some difficulty in collecting fine powder product completely, pulverized fractions were calculated from sample weights filtered out against initial loading sample weights.

2.4 Water soluble fraction measurement

20mg of Silk fibroin powder filtered with 90 micron mesh was mixed with 2 ml of distilled water and left at room temperature for 1 h. Then the solution was centrifuged to separate undissolved powder. The amount of protein dissolved in the solution was weighed after drying the solution at 105 °C for 1 h.

## 3. Results and Discussion

3.1 Effects of dose and irradiation atmosphere

Fig.1 shows the results of pulverized fractions obtained for degummed silk fiber irradiated from 250 to 1000 kGy under the atmosphere of nitrogen or oxygen. It should be noted from this figure that it is quite difficult to crush unirradiated silk fiber into powder. Pulverized fractions increased with dose. Samples irradiated in the oxygen atmosphere yielded about 60% for 250 kGy and more than 90 % for 1000 kGy. Samples irradiated in the nitrogen atmosphere was difficult to be pulverized compared with samples irradiated in the oxygen atmosphere.

Fig.2 shows the electron microscope photograph of silk fibroin powder obtained from fiber irradiated 1000 kGy in the oxygen atmosphere. There are seen fine powders in sizes less than 10 micron in diameter. It might be expected that optimization of milling conditions would yield much finer powders.

3.2 Effect of degumming

Raw silk fibers from cocoon consist of two proteins, fibroin and sericin, where the sericin surrounds two fibroin filaments in the center, playing a role of adhesive for the

two fibroin filaments and raw silk fibers. Degumming is a treatment of removing the sericin from the raw silk fibers. Fig.3 shows the effect of degumming on pulverization of irradiated silk fiber irradiated in nitrogen. Although there was little difference in pulverized fraction between raw and degummed silk fibers at higher doses, raw silk fibers shows much less pulverized fraction than degummed ones at 250 kGy.

3.3 Dissolution of silk fibroin powder into water

Fig.4 shows the amount of protein extracted by a distilled water from silk fibroin powder obtained from fiber irradiated in oxygen. As well known, unirradiated silk fibroin fiber is not soluble into water. Silk fibroin powders obtained from irradiated fiber exhibited significant dissolution into water. Soluble fractions of silk fibroin powders increased from 35% at 250 kGy to 60 % 1000 kGy. Such significant dissolution of silk fibroin powder into water had been beyond our expectation before we started this work. Molecular weight and amino acid analyses are now under way to characterize this soluble part of silk powder.

## 4. Conclusions

Pulverization behavior of silk fiber irradiated with an electron beam has been investigated in the dose range of 250 to 1000 kGy under the atmosphere of nitrogen or oxygen. The following conclusions have been drawn:

(1) It is possible to prepare silk powder directly from irradiated silk fiber only using physical crushing.

(2)Silk fiber irradiated in the oxygen atmosphere is easier to be pulverized compared to one irradiated in the nitrogen atmosphere.

(3)Raw silk fiber is less pulverized than degummed one.

(4)Silk fibroin powder has remarkable solubility in cold water from 40 to 68 % depending on dose.

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## Reference

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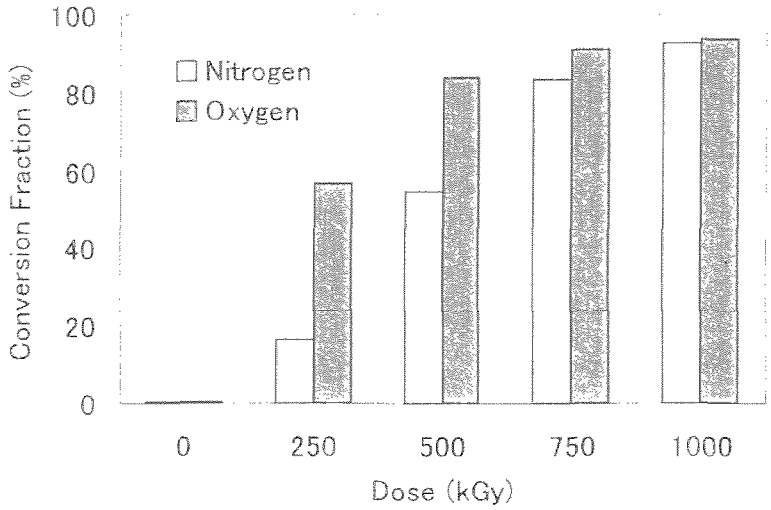


Fig.1 Pulverization of Irradiated Silk Fiber(degummed)

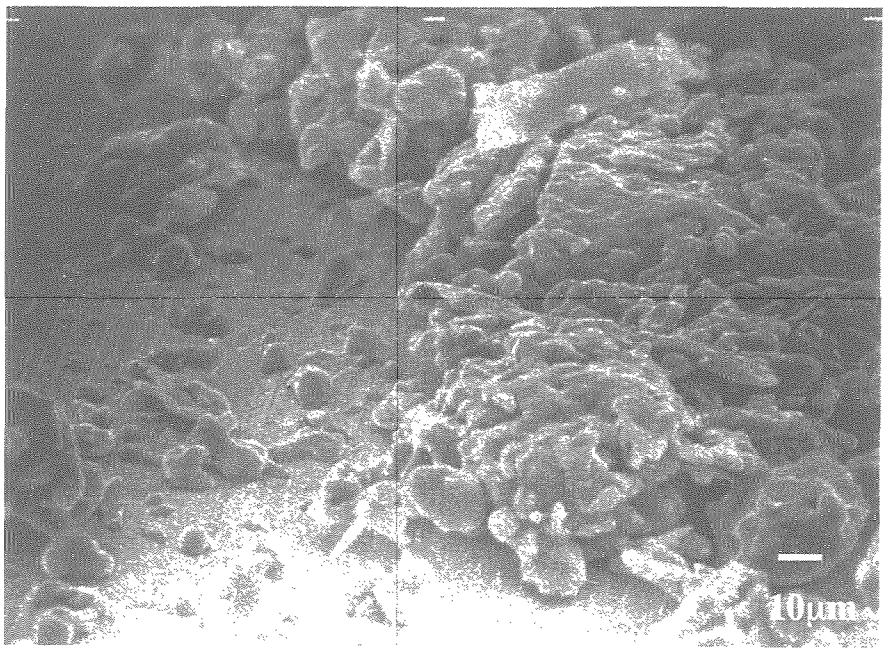


Fig.2 Photograph of silk powder(1000 kGy, Oxygen) taken with electron microscope

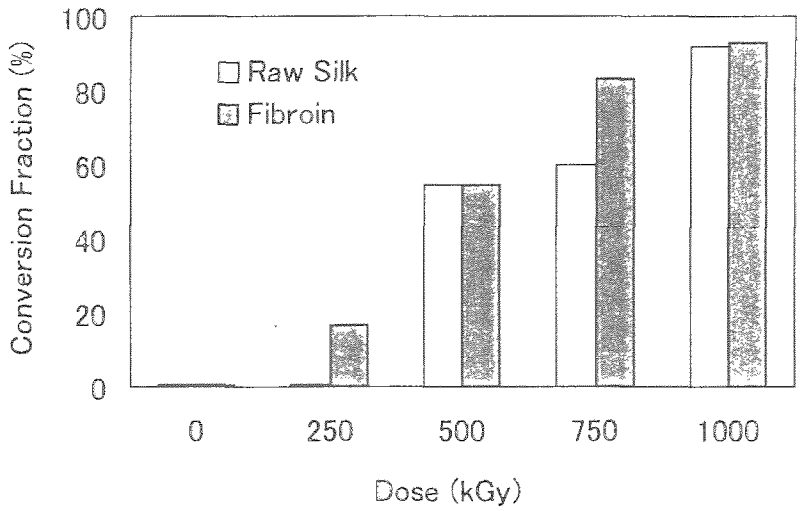


Fig.3 Comparison of pulverization behavior between raw and degummed silk fibers

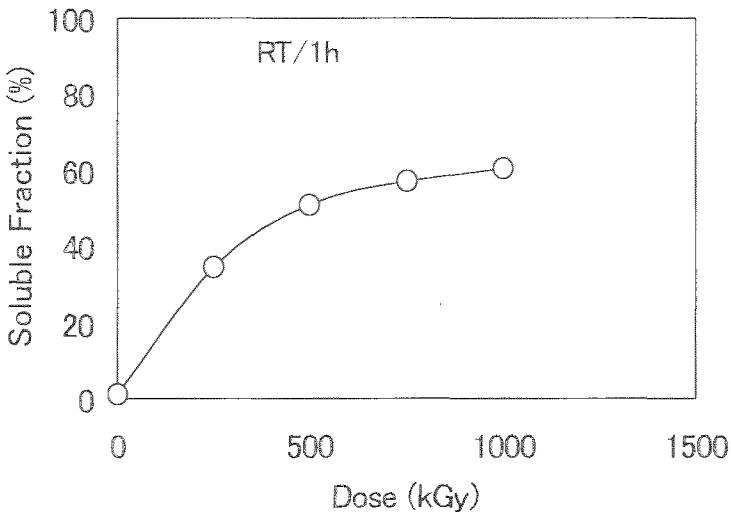


Fig.4 Dissolution of silk fibroin powder in water at room temperature.