

A Study on the Calcium and Iron Content of the *Undaria pinnatifida suringar*

Sook-Hee RHEE

Department of Home Economics, Pusan National University

미역중의 Calcium 및 Iron의 함량에 대하여

이 숙 희

부산대학교 문리과대학 가정학과

해운대 동백섬 근해에서 채취한 미역을(*Undaria pinnatifida suringar*) 유초와 성초로 구분하여 각각 부위 별로 calcium과 iron의 함량을 정량하였다.

Calcium은 과망간산칼륨 적정법에 의하였으며, iron은 o-phenanthrolin 비색법에 의하여 정량하였다.

실험결과 유초나 성초에서 calcium은 일부분에 가장 많이 함유되어 있는 경향이였으며, 일부분의 Iron 함유량은 줄기부분의 약 2배가 됨을 발견하였다.

ABSTRACT

The *Undaria pinnatifida* collected in the waters of Dongbaek Island were grouped into young and adult ones and calcium and iron contents of each were analyzed by parts, i.e., separately for blade, stripe, and sporophyll by potassium permanganate titration method and spectrophotometric method respectively (O-phenanthrolin used). The results showed that the blades contain higher percentage of calcium than other parts both in young and adult seaweeds and that proportionately twice as much iron is contained in the blades than in the stripes.

INTRODUCTION

Many studies have been undertaken to determine the chemical composition of seaweed. Ishibashi and Sahara,¹⁾ for example, made qualitative analysis of the seaweed, and Ishibashi and Yamamoto²⁾ made quantitative analysis for the ash, natrium, and kalium contents while Yamamoto³⁾ did the same for iron, copper, phosphor, and magnesium contents of the seaweeds. No attempt, however, has ever been made to investigate the chemical composition of the seaweed separately by parts and by the maturity of the seaweeds.

The author collected samples of seaweed, separated the young ones from the old, and attempted to determine the calcium and iron contents contained in different parts of the seaweed, i.e., stripes, blades, and sporophyll. The calcium contents was determined by the potassium permanganate titration method and the iron contents by the O-phenanthrolin method.^{4,5)}

METHODS

Treatment of the sample

Seaweed was collected during the months of April and May 1971 in the waters near Dongbaik Island of Haeundae and was classified into young and adult ones. The samples were at first washed lightly with the sea water at the time of collection to avoid partial dissolution of the contents and then were washed again with tap water until the chloride reaction of the water disappeared. The samples were dried at the temperature of 40° to 50°C in a dryoven, were powdered to 40 to 60 meshes, and were stored in a desiccator for later analysis.

Determination of moisture

A sample weighing 10 grams was dried at the temperature of 105°C and the moisture content was determined as the difference in weight of the sample before and after the drying process.

Determination of ash content

The residue of the process of the moisture content determination was burned to ash at the temperature of 400° to 500°C in electric furnace (5.5KVA) until it became carbon free, and then the content was determined.

Determination of calcium content

One gram of ash was dissolved into 20 ml of hydrochloric acid(1+1), was diluted to 250 ml with warm water and then was boiled with the addition of 3 or 4 drops of nitric acid. Then, 10 ml of saturated ammonium chloride and 2 or 3 drops of methylred indicator were added to the solution, and then ammonia water(1+1) was added a little by little with stirring.

When the solution began to turn from red to yellow, more ammonia water was added and boiled for another minute, after which the precipitate was filtered with 5B filter paper and then was washed with warm ammonia nitrate. The precipitate was later used for the determination of iron content.

The filtrate was concentrated by heating in a 500 ml beaker with the addition of 2 or 3 drops of methylred indicator and 20 ml of 4% ammonium oxalate. Then ammonium hydroxide was added slowly to the solution until it turned from red to yellow while the solution was being stirred and kept boiling. The resultant precipitate of calcium oxalate was kept warm in a water bath for an hour or so, was filtered with 5B filter paper, and then was washed two or three times with 0.1 percent ammonium oxalate solution.

The precipitate on the filter paper was dissolved with 20 ml of hydrochloric acid(1+1) and was diluted with water to the volume of 200 ml. Then again, 1 or 2 drops of methylred indicator and 2 ml of 4 percent ammonium oxalate were added and the whole solution was kept warm for an hour after which it was filtered.

The precipitate thus obtained was washed 8 to 10 times with cold water until the oxalic acid reaction disappeared. After adding 150 ml of warm water and 50 ml of sulfuric acid, the solution was warmed to the temperature of 70°C and was titrated with standard 1/30 N potassium permanganate solution.

Determination of iron content

The hydroxide precipitate which had been obtained at the time of calcium content determination was dissolved into 20 ml of hydrochloric acid(1+1) and then was boiled with the addition of 2 ml of nitric acid.

The precipitate thus obtained was dissolved with 5 ml of hydrochloric acid(1+3) which was boiled for 5 minutes, adjusted at PH 3.5, and then diluted with distilled water to the volume of 100 ml. Fifty ml of above solution was taken to which 1 ml of 10 percent hydroxylamine hydrochloride, 5 ml of O-phenanthroline, and 5 ml of acetate buffer were then added and diluted with distilled water to obtain a

volume of 100 ml. Then, with this sample the absorbance was measured at $500m\mu$ with spectrophotometer Q. U.3 type by comparison with the standard iron solution which had been obtained through the same process as the sample solution.

RESULTS

Moisture content

The percentage of the moisture content of various parts which was obtained by drying for two hours 10 grams of sample at the temperature of 105°C is shown in table 1. Figure 1 is a graphical representation of table 1. Figure 1 shows that the moisture is contained more in the blade than in any other parts. More in the younger one than the old, and the least in the adult stripe. As a whole the young ones contain more moisture than the old, and the sporophyll more than the stripe.

Table 1. The percentage of moisture content in the *Undaria pinnatifida*

Parts of seaweed	Classification of Sample	Moisture(%)
Adult Stripe	1st Sample	3.29
	2nd Sample	3.89
	3rd Sample	3.57
	Average	3.58
Adult Blade	1st Sample	6.35
	2nd Sample	6.99
	3rd Sample	7.43
	Average	6.92
Sporophyll	1st Sample	4.16
	2nd Sample	5.33
	3rd Sample	5.49
	Average	4.99
Young Stripe	1st Sample	6.92
	2nd Sample	6.02
	3rd Sample	7.09
	Average	6.68
Young Blade	1st Sample	9.61
	2nd Sample	10.12
	3rd Sample	10.12
	Average	9.95

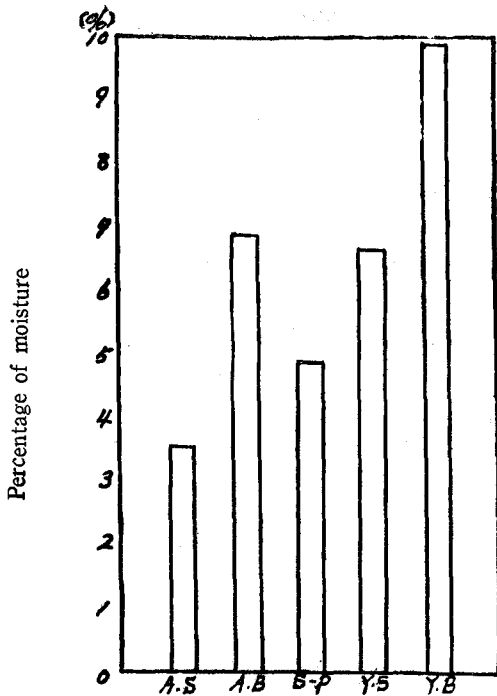
Ash content

Table 2 shows the ash content of different parts of seaweed which was determined by burning the residue of the moisture determination process at the temperature of 400° to 500°C in an electric furnace.

Figure 2 represents graphically what is contained in table 2. It is shown in figure 2 that more ash is contained in the stripes than in any other parts, that twice as much ash is found in the adult stripes than in the adult blades, and that the least is contained in the young blades. From this it may be concluded that more ash is contained in the harder parts of the *Undaria pinnatifida*.

Table 2. The percentage of ash contained in various parts of eyth oung and adult *Undaria pinnatifida*

Parts of seaweed	Classification of Sample	Time Burned(hr.)	Ash(%)
Adult Stripe	1st Sample	14	36.17
	2nd Sample	23	32.39
	3rd Sample	14	34.70
	Average	17	34.42
Adult Blade	1st Sample	14	16.13
	2nd Sample	23	13.77
	3rd Sample	14	14.78
	Average	17	14.89
Sporo phyll	1st Sample	14	18.88
	2nd Sample	23	16.43
	3rd Sample	23	16.71
	Average	20	17.34
Young Stripe	1st Sample	24	18.91
	2nd Sample	24	19.38
	3rd Sample	26	19.69
	Average	25	19.33
Young Blade	1st Sample	21	12.67
	2nd Sample	22	12.53
	3rd Sample	22	12.59
	Average	22	12.59



Parts of *Undaria pinnatifida* classified

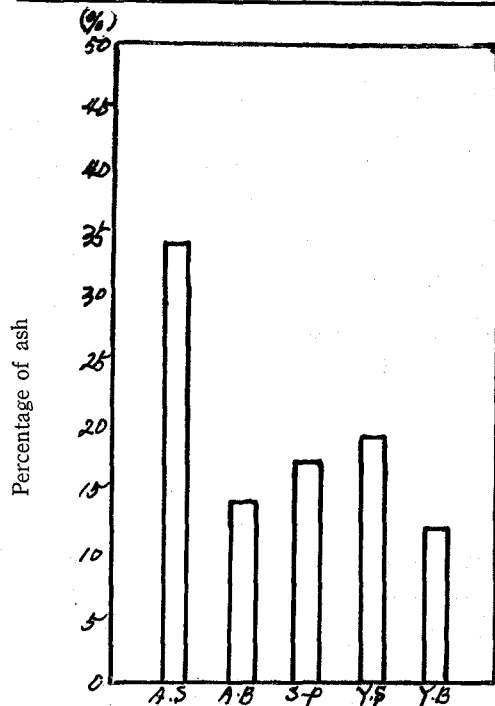
Fig. 1. The percentage of moisture content in the various parts of the *Undaria pinnatifida*.

The following abbreviations are used:

- A.S. for Adult Stripe
- A.B. for Adult Blade
- S-P for Sporophyll
- Y.S. for Young Stripe
- Y.B. for Young Blade

Calcium content

Table 3 shows the calcium content and the amount of potassium permanganate consumed in titrating 1 gram of ash by the potassium permanganate titration method. The average percentage of calcium contained in different parts of the *Undaria pinnatifida* is shown graphically in figure 3. Here, it is indicated that the blades contain more calcium than the stripes, and that the most is contained in the adult blades and the least in the sporophyll.

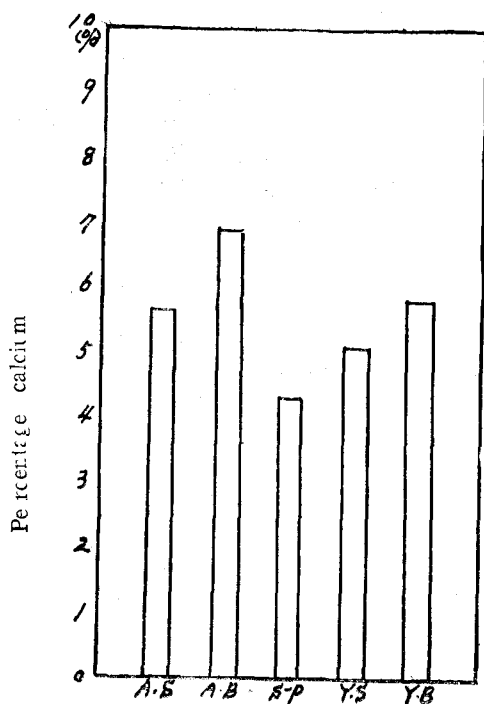


Parts of *Undaria pinnatifida* Classified

Fig. 2. The percentage of ash content in parts of the *Undaria pinnatifida*

Table 3. The KMnO₄ consumption and the percentage of calcium content in parts of the *Undaria pinnatifida* (KMnO₄, ml=0.000935 CaO)

Titration	Adult Stripe	Adult Blade	Sporo phyll	Young Stripe	Young Blade
1st	86.5 ml	101.5 ml	63 ml	75.2 ml	85.9 ml
2nd	86.2 ml	104.4 ml	64.5 ml	75.8 ml	89.5 ml
3rd	86.0 ml	102.9 ml	63.8 ml	79.45 ml	87.9 ml
Average	86.23±0.266 ml	102.9+0.966 ml	63.75±1.756 ml	76.82±1.756 ml	87.7±1.266
Calcium(%)	5.71%	6.83%	4.27%	5.13%	5.89%



Parts of *Undaria pinnatifida* classified

Fig. 3. The percentage of calcium content in parts of the *Undaria pinnatifida*.

Iron content

A set of standard iron solution of different volumes, 5, 10, 15..., 50 ml, were prepared and each of them was mixed with 1 ml of 10% hydroxylamine hydrochloride, 5 ml of O-phenanthroline, and 5 ml of acetate buffer. The solution, then, was diluted with distilled water to 100 ml and the absorbance was measured at 500 mμ, the results of which are shown in table 4.

Table 4. Absorbance of standard iron solution at 500 mμ

Volume of the standard iron solution (ml)	Absorbance(-log T)
5	0.052
10	0.106
15	0.164
20	0.232
25	0.258
30	0.309
35	0.367
40	0.420
45	0.472
50	0.520

The correlation coefficient of the amount of iron in the standard iron solution on the absorbance was found to be 0.9988, indicating a high degree of correlation between the two. To obtain a regression line which depicts the relationship between the iron quantity and the absorbance, the least-square method was used. Assuming a linear relationship of the form $Y=a+bX$ and using the normal equation of $\sum Y=N a+b \sum X$, $\sum XY=a \sum X+b \sum X^2$ constants were computed to be $Y=-0.5813+96.81X$. The values of the absorbance (X) measured of the different parts of the sample by the O-phenanthroline method at 500 mμ with spectrophotometer Q.U. 3 type were placed in the equation to obtain the value of iron content(Y) shown in table 6.

Figure 6 is a graphic version of the table 6. It can be seen that the adult *Undaria pinnatifida* contains

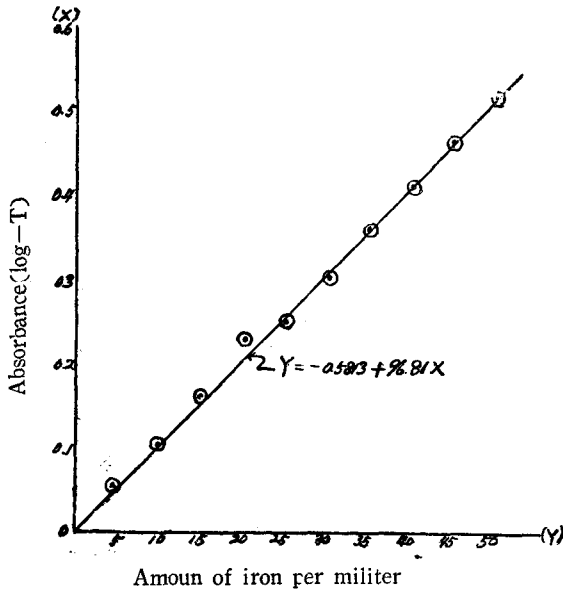


Fig. 4. Regression line of the standard iron solution

Table 5. Computation involved in determining the regression line equation by the least-square method

Amount of iron(Y)	-logT (X)	XY	X ²
5	0.053	0.265	0.0028
10	0.106	1.060	0.0112
15	0.164	2.460	0.0269
20	0.232	4.640	0.0538
25	0.258	6.450	0.0665
30	0.309	9.270	0.0955
35	0.367	12.845	0.1347
40	0.420	16.800	0.1764
45	0.472	21.240	0.2228
50	0.520	26.000	0.2704
Total	2.900	101.028	1.0610

Table 6. The percentage of iron content in parts of the *Undaria pinnatifida*

Titration	Adult stripe	Adult blade	Sporophyll	Young stripe	Young blade
1st	0.414	1.148	0.606	0.110	0.310
2nd	0.449	1.140	0.602	0.138	0.297
3rd	0.457	1.152	0.590	0.150	0.290
Average(-log T)	0.440±0.0017	1.140±0.006	0.599±0.006	0.133±0.015	0.299±0.007
Fe(r/ml)	42.015	110.363	57.989	12.876	28.946
Fe(%)	0.0042	0.0110	0.0058	0.0013	0.0029

much more iron than the young one, and the blades contain more of it than the stripes both in the young and adult ones. The adult blades contained more than twice as much iron as the adult stripes or sporophyll, and about three times as much as the young blades or stripes.

CONCLUSIONS

Samples of *Undaria pinnatifida suringar* which were collected in the waters of Dongbaik Island were analyzed to determine the ash, calcium, and iron contents of the different parts of the young and adult *Undaria pinnatifida suringar*.

The ash content was first determined by drying the sample at 40° to 50°C, making powder of it, and then burning it at 400° to 500°C in an electric furnace. The calcium content was determined by the potassium permanganate titration method and the iron content by the O-phenanthroline method.

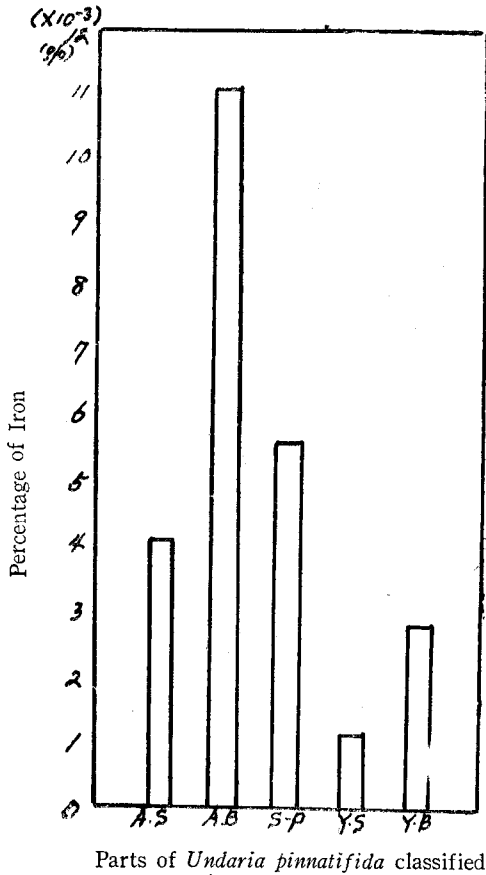


Fig. 5. The percentage of iron content in parts of *Undaria pinnatifida*.

The following results were obtained from the experiment.

The ash content in *Undaria pinnatifida* ranged from 12.59% to 34.42%, more in the adult than in the young ones, and more in the stripes than in the blades.

The calcium content was found to range from 4.27% to 6.83%, and more of it was found in the blades than in the stripes.

The iron content ranged from 0.0012% to 0.0110%, more was found in the blades than in the stripes, and the adult ones contain about three times as much iron as the young ones.

The adult ones were found to contain more calcium and iron than the young ones.

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