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Determination of Chromium Mineral in Foodstuffs in Indonesia using INAA Method

Fatmawati Nurcahyani[1,*], *Saeful* Yusuf[1], *Istanto*[1], *Siti* Suprapti[1], *Alfian*[1], *Th Rina* Mulyaningsih[1], *Ferly* Hermana[1], *Firda* Amalia[1], and *Sari Hasnah Dewi*[1]

[1]Research Center for Radiation Detection and NuclearAnalysis Technology, BRIN, KST BJ Habibie, South Tangerang, Indonesia

Abstract. The concentration of the element chromium (Cr) was determined in 76 types of food ingredients from four provinces in Indonesia. The element Cr is one of the essential elements needed by the human body for physiological processes. Although the amount needed for Cr is relatively small, this mineral is very important for the insulin hormone to function properly. Chromium deficiency can result in decreased insulin sensitivity, glucose intolerance and an increased risk of diabetes. The Cr element was determined using the Instrumental Neutron Activation Analysis (INAA) method in the GA. Siwabessy Multipurpose Research Reactor. The reactor which is located in the Puspiptek area has a thermal neutron flux of around 5.47 x 10^{13} n.cm^{-2}s^{-1}. Bombardment Cr-50 in foodstuffs samples by thermal neutrons will produce Cr-51 through the n, γ nuclear reaction. The radiosotope Cr-51. The radiosotope Cr-51 which is the result of thermal neutron activation has a half-life of 27.7 days. The single energy peak of the Cr-51 gamma-ray can be observed at 320.08 keV. The results showed that cassava leaves, melinjo leaves, watercress, sweet potato leaves, papaya leaves, ipomoea aquatica, kenikir, pumpkin leaves, spinach and mushrooms are foodstuffs rich in Cr compared to other foodstuffs. Foodstuffs from Pandeglang Regency-Banten Province have relatively higher Cr concentrations than other three locations. The concentration of Cr in foodstuffs ranges from 0.039 to 5.244 µg/g.

1 Introduction

Chromium (Cr) is one of the essential minerals needed by the human body for physiological processes [1]. Lack of Cr mineral in the body can cause mineral deficiency disease [2]. The mineral Cr is required in relatively small amounts by the body about 25 – 35 µg/day for adult, so Cr is classified as an essential micro-mineral [3]. The mineral Cr has been identified as an element that allows the hormone insulin to function correctly. Lack of the mineral Cr can cause decreased insulin sensitivity, glucose intolerance and increased risk of diabetes [2]. Mineral Cr has become popular as a dietary supplement for diabetes prevention [4]. In Indonesia, cases of diabetes mellitus continue to increase. The results of basic health research from the Ministry of Health of the Republic of Indonesia show that the prevalence of diabetes mellitus in 2018 increased by 2.6% compared to 2013. In 2018

*Corresponding author: fatm003@brin.go.id

the prevalence of diabetes mellitus sufferers aged over 15 years was 8.5% of Indonesia's population or around 14 million people [5].

In general, the intake of the mineral Cr enters the human body through food consumed daily. Although the mineral Cr deficiency is not the only cause of diabetes the knowledge about Cr content in the food consumed by the public is quite impressive. The mineral database including Cr in food ingredients is significant and needed to calculate a person's nutritional adequacy. In Indonesia, the database for the mineral concentration of Cr in various foodstuffs is still very limited. Even the table of Indonesian food composition in 2017 published by the Ministry of Health of the Republic of Indonesia does not include the Cr content in it [6]. However, several studies have been conducted to determine various essential minerals, including Cr in foodstuffs both in Indonesia and in other countries. We referred to method in the paper "Determination of micro essential element Fe in foodstuffs using instrumental neutron activation analysis (INAA)" [7][8][9][10][11]**.**

Neutron activation analysis method (AAN) is a method of nuclear analysis technique that has high accuracy and sensitivity [12][13]. This method is often used to determine the mineral concentration in foods that have a relatively small concentration with the ppm level [14][15][16] [17][18] Research on Cr and Zn content in foodstuffs in Lebak and Tangerang Regencies, Banten Province, has been conducted by Saeful [19].

This study aims to determine concentration of Cr in various foodstuffs commonly consumed by the population in Indonesia. Food samples came from traditional markets in Pandeglang Regency-Banten Province, Cianjur Regency-West Java Province, Magelang Regency-Central Java Province and Bangkalan Regency - East Java Province. The four provinces are located on the island of Java, which has a population of 150.4 million or 56.3% of Indonesia's population in 2019. Determination of the elemental content of Cr was carried out using the Nuclear Analysis Technique, namely the neutron activation analysis method. Compared to previous studies, the types of samples tested in this study were more varied and covered more locations. The neutron source used comes from Siwabessy-multipurpose research reactor in the Puspiptek Serpong area. The mineral content is calculated based on the dry weight of the food portion that is usually consumed. This database of Cr mineral content for various foodstuffs can complement the database of foods in Indonesia and is useful for stakeholders.

2 Methods

2.1 Sampling and sample preparation

Foodstuffs samples were taken from markets in Pandeglang Regency-Banten Province, Cianjur Regency-West Java Province, Magelang Regency-Central Java Province and Bangkalan Regency-East Java Province. The food material used as the sample is part of the food ingredients generally consumed by the people of Indonesia. Such as Vegetables, Fish, Fruits, beans. We take about 100 gram sample then the sample was washed and then sliced into small pieces and put in the freezer box. Then the foodstuff sample is dried using a freeze dryer equipment at a temperature of -90 ºC and a pressure of 0.086 mbar for 5 days. After drying, the sample is crushed in a blender using a titanium knife or pounded with a mortar to a size of about 100 mesh. The sample is ready to be tested by the neutron activation analysis (NAA) method.

2.2 Sample preparation for irradiation

A total of 50-100 mg of sample was weighed with a micro-electric balance, put into a vial low density poly ethylene (LDPE) and the vial was tied with heating. The samples, standard and SRM were wrapped in aluminum foil and arranged in the same layer, covered with a "rabbit capsule" made of aluminum. The targets were irradiated for 3 hours at the hydraulic rabbit system facility in Siwabessy-multipurpose research reactor. The target irradiation was carried out at a reactor power of 15 MW and a thermal neutron flux of about 5.47 x 10^{13} n. $cm^{-2}s^{-1}$. The nuclear reaction that occurs in determining Cr by the NAA method is Cr-50 (n. γ) Cr-51 [13].

2.3 Data acquisition

Post-irradiation testing of samples was carried out after 18 days of radioactive sample decay. Decay aims to reduce the effects of medium and short half-life radionuclides that can interfere with the peak determination of the Cr-51 isotope. Cr-51 has t½ = 27.7 days and emits gamma rays at an energy of 320.08 keV. The sample, standard and SRM were measured for 2 hours using a high-resolution pure germanium detector made in Canberra with the aid of Genie- 2000 software.

2.4 Performance test of NAA method Data

Proving the performance of the NAA method was carried out by determining the concentration of Cr in standard reference materials from the National Institute of Standard and Technology, namely SRM 1573a (tomato leaves). NIST SRM-2976 (trace element and methylmercury in mussel-tissue) was used as standard. The Cr concentration produced in this study was compared with the Cr concentration from the certificate. Furthermore, several statistical calculations are carried out such as relative deviation, the ratio of results to the certificate value and the u-test score. From the results of these statistical calculations can be concluded that the performance of the NAA method for determining the concentration of Cr.

2.5 Chromium concentration calculations

Elemental Cr concentrations were calculated according to the comparison NAA method. The calculation of element concentration using the comparative NAA method follows the following equation:

$$C_X = \frac{A_X.D_X.W_S.C_S}{A_S.D_S.W_X} \tag{1}$$

where
C_x : concentration of Cr in the sample
C_s : concentration of Cr in the standard
A_x : count rate of Cr-51 in the sample
A_s : count rate of Cr-51 in the standard
D_x : decay correction of sample
D_s : decay correction of standard
W_x : mass of sample
W_s : mass of standard

3 Result and Discussion

The results of the determination of the Cr concentration in the standard reference materials of NIST SRM 1573a and the Cr concentration according to the certificate are shown in Table 1. Table 1 also shows the relative deviation, the ratio of the results to the certificate and the u_{-test} score.

Table 1. The performance of the NAA method in determining Cr in NIST SRM 1573a.

SRM	This Result Cr (µg/g)	Certified Cr (µg/g)	Relative Deviation σ (%)	Ratio of result to certified R	U_{-test} Score
SRM 1573a	1.86±0.10	1.99±0.06	(6.3)	0.94	1.08

Calculation of the relative deviation σ, ratio and U-test score with the following equation:

in Table 2 should be used.

$$\sigma = \frac{\text{result-certificate}}{\text{certificate}} \times 100\% \tag{2}$$

$$\text{ratio} = \frac{\text{result}}{\text{certificate}} \tag{3}$$

$$u_{test} = \frac{|\text{certificate-result}|}{\sqrt{unc^2_{certificate} + unc^2_{result}}} \tag{4}$$

where
result : Cr concentration obtained from testing
certificate : Cr concentration of the certificate
$unc_{certificate}$: the uncertainty of the certificate
unc_{result} : the uncertainty of the testing

U-test scores for the determination of Cr in the SRM 1573a were 1.08. The relative deviation and ratio of the results to the certificate value in the determination of Cr in the SRM NIST 1573a were -6.3% and 0.94, respectively. Based on the u-test score criteria, as shown in table-2, the two u-test scores are at u <1.64 with the status 'reported results are not significantly different from the certificate value'. The results above indicate that the NAA method to be used in this study has a good performance for determining the Cr concentration.

Table 2. Assessment criteria U-test Score.

U-test Score	Status
u<1.64	The results reported were not significantly different from the value of the certificate
1.95>u>1.64	The reported results may not be significantly different from the value of the certificate
2.58>u>1.95	There is no apparent difference between the reported results and the certificate value
3.29>u>2.58	The reported results may differ significantly from the value of the certificate

u>3.29	The results reported were significantly different from the value of the certificate

Table 3 shows the results of determining the concentration of Cr elements in various foodstuffs from Pandeglang Regency-Banten Province, Cianjur Regency-West Java Province, Magelang Regency-Central Java Province and Bangkalan Regency-East Java Province. In this research, the determination of Cr mineral has been successfully carried out in 76 types of samples from 4 provinces in Java-Indonesia. The concentration of Cr mineral in foodstuffs is expressed in mg of mineral Cr in 1 kg of dry sample. Some of the results in Table-3 show the value of N which means that the Cr mineral is not determined because the sample cannot be collected when sampling. The Cr concentration in the foodstuff ranges from 0.039 to 5.244 [μg/g]. The Cr concentration of each type of food is different for each region. This is understandable because these foods depend on different soil fertility in each location. The interesting thing is the tendency of foodstuffs to have the same concentration level in each location, for example cassava leaves have a relatively high concentration compared to other food ingredients. Based on the data in table 3, it is found that cassava leaves, melinjo leaves, watercress, sweet potato leaves, papaya leaves, ipomoea aquatica, kenikir leaves, pumpkin leaves, spinach and mushrooms are foods that are rich in Cr elements compared to other foods.

Table 3. The concentration of Cr mineral in foodstuff in Indonesia (μg/g dry weiht).

No.	**Foodstuff**	**Cianjur regency**		**Magelang regency**		**Bangkalan regency**		**Pandeglang regency**		**Average**
		c	**unc**	**c**	**unc**	**c**	**unc**	**c**	**unc**	**(μg/g)**
1	*Manihot esculenta* leaves	2.483	0.141	2.886	0.166	N		5.244	0.376	3.538
2	*Gnetum gnemon* leaves	1.494	0.086	2.801	0.172	N		4.969	0.396	3.088
3	*Nasturtium officinale*	3.052	0.172	3.359	0.203	N		1.628	0.095	2.680
4	*Ipomoea batatas* leaves	N		1.664	0.098	N		3.598	0.241	2.631
5	*Carica papaya L.* leaves	1.544	0.087	N		1.412	0.083	4.881	0.371	2.612
6	*Ipomoea aquatica*	1.491	0.083	N		1.126	0.069	4.252	0.309	2.290
7	*Cosmos* leaves	N		2.763	0.159	1.545	0.092	N		2.154
8	*Cucurbita moschata* leaves	1.567	0.090	2.365	0.137	N		N		1.966
9	*Amaranthus tricolor (bayam cabut)*	1.351	0.078	N		1.075	0.063	3.068	0.235	1.831

10	*Pleurotus ostreatus (Jamur tiram)*	N		N		0.460	0.030	3.148	0.250	1.804
11	Brown rice	0.816	0.050	1.487	0.089	2.806	0.179	N		1.703
12	*Brassica chinensis var. parachinensis*	2,027	0,113	N		2,228	0,119	0,788	0,097	1.681
13	*Momordica charantia*	1.829	0.105	1.230	0.071	0.845	0.054	2.682	0.181	1.647
14	*Sechium edule*	0.893	0.051	1.946	0.113	N		2.021	0.139	1.620
15	*Brassica rapa*	2.717	0.157	1.770	0.111	1.540	0.080	0.419	0.048	1.612
16	*Brassica oleracea var. botrytis*	1.281	0.074	N		0.890	0.054	2.535	0.204	1.569
17	*Psophocarpus tetragonolobus*	1.440	0.079	N		1.626	0.100	N		1.533
18	*Auricularia auricula-judae*	1.044	0.060	N		N		2.006	0.134	1.525
19	*Limnocharis flava* leaves	2.727	0.152	N		N		0.189	0.036	1.458
20	*Brassica oleracea var. italica*	1.420	0.090	1.279	0.076	1.647	0.096	N		1.449
21	Sauropus androgynus	1.888	0.108	N		2.296	0.129	0.129	0.024	1.438
22	*Musa paradisiaca* heart	1.800	0.105	0.931	0.061	1.454	0.083	N		1.395
23	*Loligo*	1.736	0.104	1.799	0.097	0.640	0.040	N		1.392
24	*Apium graveolens*	1.182	0.065	1.673	0.101	1.226	0.080	N		1.360
25	Banana heart	1.173	0.068	1.536	0.107	N		N		1.355
26	*Solanum melongena* (round)	1.287	0.068	1.910	0.111	N		0.287	0.035	1.161
27	Allium fistulosum	1.418	0.080	1.196	0.073	0.815	0.059	N		1.143
28	*Solanum melongena*	N		N		1.155	0.069	1.103	0.064	1.129

29	*Brassica rapa subsp. chinensis*	2.364	0.134	0.717	0.042	1.253	0.083	0.164	0.029	1.125
30	Young *Zea Mays*	1.558	0.088	1.419	0.081	N		0.396	0.080	1.124
31	*Carica papaya L.*	N		1.611	0.095	0.619	0.034	N		1.115
32	*Rastrelliger faughni* (kembung)	0.898	0.054	1.183	0.067	N		N		1.041
33	*Luffa acutangula*	1.091	0.063	1.252	0.072	1.580	0.100	0.072	0.015	0.999
34	Tempeh	0.698	0.043	1.140	0.067	0.970	0.060	N		0.936
35	Phaseolus vulgaris	0.775	0.046	N		1.090	0.070	N		0.933
36	Cucumis sativus	0.851	0.048	N		0.980	0.060	N		0.916
37	*Daucus carota*	1.171	0.067	0.960	0.056	0.598	0.038	N		0.910
38	*Gnetum gnemon skin*	1.133	0.074	1.326	0.077	0.780	0.040	0.381	0.134	0.905
39	Pisum sativum var. saccharatum	0.856	0.050	0.941	0.058	N		N		0.899
40	Bean sprouts	0.646	0.038	0.487	0.034	0.432	0.028	1.963	0.156	0.882
41	*Cyprinus carpio*	0.705	0.041	1.029	0.059	N		N		0.867
42	Raw Artocarpus heterophyllus	1.508	0.086	N		0.963	0.058	0.078	0.010	0.850
43	*Lutjanus campechanus*	1.028	0.064	N		0.631	0.037	N		0.830
44	Chanos chanos	0.681	0.039	0.968	0.055	0.620	0.050	0.864	0.051	0.783
45	Zea Mays	0.772	0.043	0.847	0.049	0.671	0.039	0.805	0.072	0.774
46	Osphronemus goramy	N		N		0.660	0.040	0.884	0.051	0.772
47	Little Thunnini *(Tuna)*	0.521	0.030	1.325	0.075	0.370	0.030	N		0.739

48	Caridea	0.652	0.037	0.878	0.048	0.610	0.040	N		0.713
49	Gallus gallus domesticus	0.612	0.034	0.981	0.053	0.530	0.040	N		0.708
50	Tofu	0.896	0.057	1.081	0.058	0.542	0.038	0.292	0.050	0.703
51	Beef	0.715	0.039	0.781	0.042	0.536	0.031	0.754	0.042	0.697
52	Cassava	0.384	0.026	N		0.345	0.020	1.322	0.107	0.684
53	Yellow sweet potato	N		0.808	0.046	N		0.554	0.051	0.681
54	White rice	0.492	0.037	0.533	0.031	1.328	0.084	0.362	0.032	0.679
55	Black sticky rice	0.489	0.033	0.972	0.058	0.521	0.033	N		0.661
56	Cat fish	N		0.653	0.038	N		N		0.653
57	Tilapia fish	N		0.551	0.030	0.380	0.030	0.985	0.063	0.639
58	Soybeans	0.641	0.037	1.010	0.069	0.168	0.059	0.725	0.042	0.636
59	White sweet potato	0.709	0.049	0.698	0.042	0.500	0.030	N		0.636
60	Lagenaria siceraria	0.528	0.030	N		0.730	0.040	N		0.629
61	Solanum sect. Lycopersicon	0.727	0.042	1.077	0.061	0.644	0.041	0.062	0.010	0.628
62	Epinephelinae	0.589	0.033	0.710	0.039	0.455	0.026	0.702	0.040	0.614
63	Aci noodle	0.358	0.022	N		N		0.772	0.067	0.565
64	Arachis hypogaea	0.729	0.040	0.849	0.050	0.104	0.017	0.500	0.054	0.546
65	Seriola lalandi	0.552	0.035	N		0.530	0.040	N		0.541
66	Brassica oleracea var. capitata	0.818	0.044	N		0.475	0.033	0.330	0.051	0.541

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67	Vigna angularis	0.399	0.028	0.558	0.034	N		0.599	0.041	0.519
68	Colocasia esculenta	0.381	0.025	0.618	0.038	N		N		0.500
69	*Archidendron pauciflorum*	0.513	0.030	0.335	0.021	N		0.640	0.037	0.496
70	Vigna radiata	0.423	0.026	N		N		0.520	0.030	0.472
71	Vigna unguiculata	0.366	0.028	0.625	0.039	N		0.361	0.027	0.451
72	Vigna unguiculata ssp. sesquipedalis	0.523	0.030	N		0.637	0.040	0.185	0.042	0.448
73	Ipomoea batatas	0.692	0.046	N		0.192	0.017	N		0.442
74	Oryza sativa glutinosa	0.466	0.025	0.646	0.039	0.083	0.012	N		0.398
75	Corn vermicelli	0.308	0.020	0.473	0.027	0.323	0.020	0.404	0.033	0.377
76	Solanum tuberosum	0.502	0.027	0.201	0.011	0.039	0.012	0.521	0.047	0.316

Note:
*N = not determined
c (concentration) and unc (uncertainty) are expressed in units of (µg/g dry weigt)

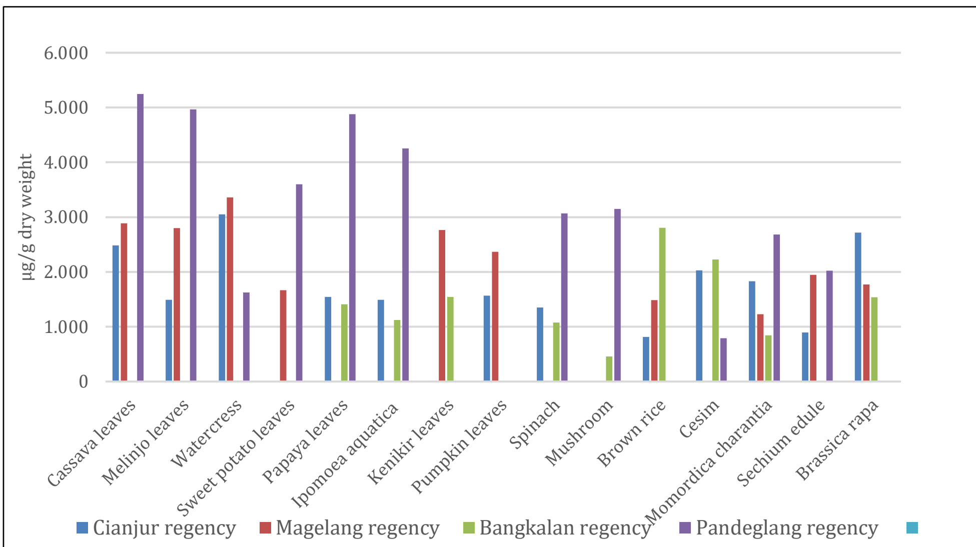

Figure 1. The concentration of Cr mineral in 15 types of foodstuffs at four different locations.

Figure 1 shows the concentration of Cr mineral in 15 types of foodstuffs from 4 different locations. In general, specific areas tend to have higher mineral concentrations than others. The data above shows that food ingredients taken from Pandeglang Regency, Banten Province, have a relatively higher Cr mineral concentration than other locations. The next area that has a relatively high concentration of Cr mineral is Magelang Regency Central Java Province.
The results of this study can be compared with the results of previous studies. Figure 2 shows the Cr concentrations of three different regions for several foodstuffs. The results obtained in this study were not much different from the results of previous studies in the Tangerang District, Banten Province. However, if the results of this study are compared to the Algerian region, the Cr concentration in foodstuffs in Algeria is higher than in Indonesia. As previously explained, this is due to differences in the fertility of the soil where the food is grown.

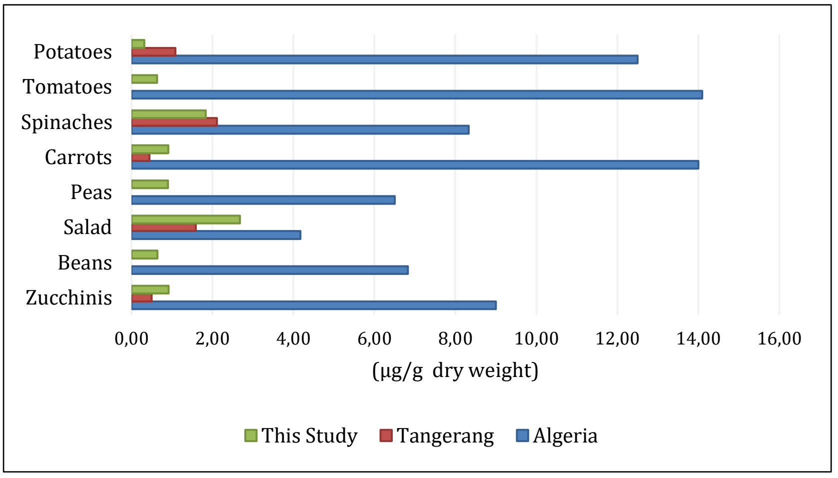

Figure 2. Comparison of Cr concentration from three regions for several foodstuffs [19][20].

Data on the Cr content of 76 types of food from 4 locations in Indonesia can complement the current Indonesian food composition table. This data is expected to be of benefit to the community and can be a reference for nutritionists in determining food formulas that are suitable for consumption, especially for diabetes prevention. However, there is a weakness from the data mentioned above, because it only displays data on Cr content in the dry sample weight. The data will be more useful if the Cr content in the value of the raw material is also displayed.
Table 4 shows an adequate intake (AI) of chromium for people of various ages. The tolerable upper intake level (UL) is the highest level of daily nutrient intake that is likely to pose no risk of adverse health effects for almost all individuals. The UL is not meant to apply to individuals who are receiving chromium under medical supervision. The toxicity of chromium differs widely depending on the valence state. This review is limited to evaluating trivalent chromium (III) because this is the main form of chromium found in food and supplements. Hexavalent chromium (VI), which has a much higher level of toxicity than trivalent chromium, is not found in food. Ingested chromium III has a low level of toxicity which is due, partially, to its very poor absorption. Several studies have demonstrated the safety of large doses of chromium (III) [4].

Table 4. Chromium adequate intake for people of various ages [4].

Ages	Cr adequate intake (AI for women)	Cr adequate intake (for men)
0-6 months	0.2 μg/day	0.2 μg/day
7-12 months	5.5 μg/day	5.5 μg/day
1-3 years	11 μg/day	11 μg/day
4-8 years	15 μg/day	15 μg/day
9-13 years	21 μg/day	25 μg/day
14-18 years	24 μg/day	35 μg/day
19-30 years	25 μg/day	35 μg/day
31-50 years	25 μg/day	35 μg/day
51-70 years	20 μg/day	30 μg/day
>70 years	20 μg/day	30 μg/day

4 Conclusion

The NAA method used in this study performed well for the determination of Cr concentration. Data on the Cr content of 76 types of food from 4 locations in Indonesia can complement the current Indonesian food composition table. The data shows that foodstuffs taken from Pandeglang Regency, Banten Province, have a relatively higher Cr mineral concentration than other locations. Based on the average mineral concentration of Cr: cassava leaves, *melinjo* leaves, watercress, sweet potato leaves, papaya leaves, ipomoea aquatica, *kenikir*, pumpkin leaves, spinach and mushrooms are food ingredients that are rich in Cr elements compared to other food ingredients. Based on Average Concentration, The need for chromium for adult can be met by consuming about 10 - 15 grams of cassava leaves per day, assuming the shrinkage of the material after drying is 30%.

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