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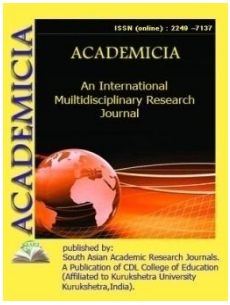

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DETERMINATION OF IODINE CONTENT IN SOME MEDICINAL PLANTS THROUGH POTENTIOMETRIC AND IODOMETRIC TITRATION

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ABSTRACT

The article provides information on the determination of the iodine content of in species of Diospyros kaki (PCh) persimmon chocolate, Diospyros kaki (KH) Korolyok-Hyakume persimmons, the extracts of Cucurbita pepo L – pumpkin fruits and Exocarpium Citri L-lemon peel, which are grown in the conditions of Andijan region, through potentiometric and iodometric titration methods.

KEYWORDS: *Persimmon, The Extracts Of Lemon Peel And Pumpkin Fruit, Potentiometric And Iodometric Titration, Electromotive Force – EF, Increase Of Potential After Equivalence Point, Starch Indicator Solution, Titration Graph, Approximate Titration, Exact Titration.*

INTRODUCTION

Although synthetic drugs containing iodine are used in thyroid disease, they can adversely affect certain systems in the human body, such as the gastrointestinal tract in patients with pathology of the gastrointestinal tract. As a result, iodine deficiency, which leads to thyroid dysfunction due to poor absorption of iodine in the body, is not eliminated [1; p. 19-23].

Given the side effects of synthetic drugs on the human body, we recommend that patients with thyroid disease use drugs made from various medicinal plants or iodine-containing food supplements. Such medicinal food supplements are used in practice in the treatment of thyroid disease and are highly effective [1; p. 19-23].

Iodine medicinal food additives are prepared from iodine-containing medicinal plants, or enriched with natural biodiesel, depending on the beneficial properties of the plant for the human body. In order to prepare medicinal food compounds containing iodine, the amount of iodine in

medicinal plants is first checked. Potentiometric and iodometric titration methods can be used to determine the amount of iodine in solutions of these plant extracts [2; p. 24-26].

The potentiometric analysis includes methods for determining the concentration of ions in a solution based on the measurement of the potential difference (electromotive force - EF) between the electrodes immersed in the test solution.

Their oxidometric capacity was used for the potentiometric determination of iodine in the analyzed solutions. Since the iodine ion is electroactive in an aqueous solution under given conditions, the oxidation-reduction pair is formed by titrant in order to determine the endpoint, ie 1 drop of 0,1 N KI solution is added to the solution to form IO_3^- / I^- pair.

$KIO_3 + 3H_2SO_4 + 5KI \rightarrow 3K_2SO_4 + 3I_2 \downarrow + 3H_2O$

In doing so, I_2 falls into precipitate. The solubility of the resulting precipitate is small (0.28 g / l).

$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$

When the titration of I_2 in the solution was completed, the last added 1 drop of $Na_2S_2O_3$ led to an increase in the electrode potential. The rise of the potential after the equivalence point can be explained using the formula [3; p. 10-20].

$$E = 1{,}33 + \frac{0{,}059}{6} lg \frac{[IO_3^-][H^+]^{14}}{[I^-]^2}$$

To assess the accuracy of the results of potentiometric determinations, we also directly determined the amount of iodine in persimmons, pumpkins and lemon peel by iodometric titration.

The method is based on the interaction of potassium iodate with potassium iodide in an acidic environment (1) and titration of the released iodine with sodium thiosulfate (2) [3; p. 10-20].

1) $KIO_3 + 5KI + 3H_2SO_4 \rightarrow 3I_2 + 3K_2SO_4 + 3H_2O$

2) $2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$

The amount of iodine in mg in the solutions obtained for analysis was calculated according to the following formula:

$$\chi = \frac{N_t * V_t * E_a * V_{er}}{1000 \; V_{ek}}$$

Here, N_t – titrant normality;

V_t – total volume of titrant consumed, ml;

E_a – equivalent molar mass of iodine compound in the test solution;

V_{er} – the mass of the sample to be tested, mg;

V_{ek} – the volume of titrant solution consumed at equivalent point, ml.

EXPERIMENTAL PART

Samples of Diospyros kaki (PCh) palm chocolate, Diospyros kaki (KH) Korolyok-Hiakume palm varieties, Cucurbita pepo L - pumpkin fruits and Exocarpium Citri L-lemon peel solution of lemon peel grown in the Andijan region as the object of analysis for the research work.

2 grams of persimmons, lemons and pumpkins weighing to the nearest 0,01 g were placed in three separate porcelain crucibles, each containing 3 ml of 20% potassium carbonate and 2 ml of distilled water, moistened, the crucible lids closed and left at room temperature for 16 h. The crucibles were then placed in a sand bath with the lid open and heated to 150–250 °C until the smoke stopped. The muffles were then placed in the muffle furnace with the lid closed and kept at 250 ℃ for 30 min. The temperature in the crucible samples was gradually raised to 500–550 ℃ until complete combustion. The firing process was continued until a white residue remained in the crucibles. A solution of 2 N sulfuric acid was added to the obtained white ash until pH = 2, and distilled water was added to them so that the volume of the solution was 100 ml. Due to the slight turbidity in the resulting solutions, the solutions were centrifuged for 10 min and separated from the precipitate [4; p. 30-33].

Potentiometric determination of iodine. Iodine in solutions of palm, lemon, and pumpkin samples prepared for analysis was separated from 50 ml of the solutions obtained for potentiometric testing. Potentiometric titration of I-ions in the obtained solutions determined the amount of iodine in the solution. I-130 ionometer was used before potentiometric determination of iodine was initiated. Initially, the filtrate from the palm sample was separated from 10 ml using a 10 ml graduated pipette and placed in a 50 ml titration beaker. To the solution was added 1 ml of 2 n H_2SO_4 solution, stirred by adding 10 ml of 0,1 N KI solution, then cover the container with a stopper and place in a dark place for 10 minutes. Add 0,01 N $Na_2S_2O_3$ from the burette to the mixture, stirring until the colour turns light yellow to the orange test solution. Approximately 2 ml of starch indicator solution was added to detect free iodine in the test solution. As a result, the mixture turned brown and the titration was continued with 0,01 N $Na_2S_2O_3$ solution until the final colour of the solution disappeared. The Pt indicator and silver chloride comparison electrodes, as well as the steel piece sterile coated completely with polyethene, were washed and dissolved in distilled water. The solution in the beaker was placed in an MM-3M magnetic mixer with electrodes and a magnetic stirrer rod. A constant rotation speed of the magnetic stirrer was set to keep the solution stirring continuously during the titration. The main attention was paid to the fact that the solution does not scatter and no gaps are formed around the electrode. The titration process was continued until the brownish colour of the solution disappeared [4; p. 30-33].

In this process, the ionomer potential was measured and the titration was performed by adding titrant fractions every 40-60 seconds from a standard solution of $Na_2S_2O_3$ in a micro burette with a volume of 25 ml. Since the amount of iodine in the test filtrate was very low, the titration was carried out by adding the titrant fractions dropwise.

40-60 seconds after the addition of each fraction (drop) of titrant, the ionomer index was expected to remain constant, and these potential values were recorded in the titration protocol. The titrant volume up to the drop corresponding to the largest potential-jump was taken as the volume corresponding to the equivalence point. The amount of iodine was calculated based on

the results of 6-7 repetitive experiments conducted in parallel. After each titration, the used vessels and electrodes were washed with distilled water.

The volume of sodium thiosulfate corresponding to the potential jump in the initial titration was approximated, and in subsequent parallel titrations, the titrant was added dropwise in the jump area. In the approximate titration, 1 ml of titrant was added [4; p. 30-33].

The point of slow jump of the potential corresponds to the equivalent point. To find the volume of titrant used for titration, the volume of titrant added during the drop titration was added to the volume of titrant (e.g., 4 ml) that went to the potential jump in the approximate titration. To find the volume of titrant used for droplet titration, the number of drops was multiplied by the volume of one drop.

To find the volume of one drop, the number of drops in 1 or 2 ml of solution was determined and the corresponding volume was divided by the number of drops.

Potentiometric titration of iodine in solutions of lemon and pumpkin fruits was carried out in the same way.

The iodometric titration method was used to estimate the potentiometrically determined amount of iodine in persimmon, lemon, and pumpkin fruit extracts.

Dissolve 10 ml of the test sample obtained for analysis in a conical flask with a volume of 250 ml in 100 ml of distilled water to the measured volume. As the resulting solution was turbid, it was filtered. To the resulting solution was added 1 ml of 2 N H_2SO_4 solution, stirred by adding 5 ml of 10% KI solution, cover the container with a stopper and leave in a dark place for 10 minutes. Because before titration, an additional process under the influence of light in the reaction mixture, oxidation of iodide ions to iodine can occur [5; p. 102-105].

$Na_2S_2O_3$ with 0,01 N from the burette was added to the test solution, stirring until the orange solution turned light yellow. When approximately 2 ml of starch indicator solution was added to the test solution, the mixture turned brownish blue. The titration was continued until the final colour of the solution disappeared.

Given the decrease in the accuracy of the analysis when using a completely uncooled starch solution, the solution was titrated while cooled. An iodine starch complex is formed which reacts very slowly when the indicator solution is added too early, which leads to high results of the analysis.

The reaction was carried out at a moderate room temperature of 25 ^oC so that the indicator solution did not lose its sensitivity when heated due to the increased volatility of iodine [5; p. 102-105].

RESULTS AND THEIR DISCUSSION

A potentiometric titration method was used to determine the amount of iodine in solutions made from dates, lemon peel and pumpkin fruits. The results of the exact and approximate titrations are presented in Table 1.

As can be seen from the results in the table, the abrupt jump in EF corresponded to a titrant area of approximately 5 ml in the approximate titration and 7 ml in the exact titration. After adding 4

ml of titrant in the approximate titration and 6 ml in the exact titration, we started dripping titration to find the exact equivalent point.

In the potentiometric titration process, the titrant consumption at the equivalence point was 5 ml and the total titrant consumption was 14 ml when titrated with sodium thiosulfate with the addition of 15 ml of distilled water to 10 ml of the persimmon extract solution.

The following equation for the amount of iodine in persimmon extract was determined on the basis of the following formula, taking into account that the iodine content of the persimmon solution tested for normality of the titrant used in the experiment was 0,01 n and the equivalent of potassium iodate was 214

$\chi = \frac{N_{Na_2S_2O_3} V_{Na_2S_2O_3} Э_{KJO_3} V_{er}}{1000\ V_{ek}} = \frac{0{,}01*14*214*10}{1000*5} = \mathbf{0,0599}$ mg $= \mathbf{59,9}$ mkg was obtained.

To carry out potentiometric titration with the addition of 15 ml of distilled water to a volume of 10 ml of a solution of lemon peel extract, 15 ml of a 0,0001 N solution of sodium thiosulfate obtained as a titrant was used.

The volume of titrant consumed at the equivalent point was 6 ml, and if we determine the amount of iodine in it, $\chi = \frac{N_{Na_2S_2O_3} V_{Na_2S_2O_3} Э_{KJO_3} V_{er}}{1000\ V_{ek}} = \frac{0{,}0001*15*214*10}{1000*6} = \mathbf{0,00053}$ mg $=$ $\mathbf{0,53}$ mkg was obtained.

TABLE 1 RESULTS OF POTENTIOMETRIC TITRATION

Persimmon extracts						***Lemon peel extract***						***Pumpkin extract***					
Approximate titration			**Exact titration**			**Approximate titration**			**Exact titration**			**Approximate titration**			**Exact titration**		
Added titrant volume, ml	Measured EYuK values, mV	The difference in EF values	The volume of titrant added, (drops) ml	Measured EYuK values, mV	The difference in EF values	Added titrant volume, ml	Measured EYuK values, mV	The difference in EF values	The volume of titrant added, (drops) ml	Measured EYuK values, mV	The difference in EF values	Added titrant volume, ml	Measured EYuK values, mV	The difference in EF values	The volume of titrant added, (drops) ml	Measured EYuK values, mV	The difference in EF values
1	400	-	1	400	-	1	400	-	1	400	-	1	400	-	1	400	-
2	400	-	2	400	-	2	400	-	2	400	-	2	400	-	2	400	-
3	398	0,02	3	398	0,02	3	398	0,02	3	398	0,02	3	398	0,02	3	398	0,02
4	394	0,04	4	396	0,02	4	394	0,04	4	396	0,02	**4**	**284**	**1,14**	4	396	0,02
5	**22**	**1,7**	5	39	0,0	**5**	**28**	**1,1**	5	39	0,0	5	258	0,2	**5**	**28**	**1,1**

	0	**4**		0	6		**0**	**4**		0	6			6		**0**	**6**
6	200	0,20	6	380	0,10	6	260	0,20	**6**	**274**	**1,16**	6	234	0,24	6	254	0,26
7	184	0,16	**7**	**258**	**1,22**	7	244	0,16	7	258	0,16	7	212	0,22	7	230	0,24
8	170	0,14	8	246	0,12	8	230	0,14	8	246	0,12	8	192	0,20	8	208	0,22
9	158	0,12	9	236	0,10	9	220	0,10	9	236	0,10	9	174	0,18	9	188	0,20
10	148	0,10	10	228	0,08	10	212	0,08	10	228	0,08	10	158	0,16	10	170	0,18
11	140	0,08	11	222	0,06	11	206	0,06	11	222	0,06	11	144	0,14	11	154	0,16
12	134	0,06	12	218	0,04	12	202	0,04	12	218	0,04	12	132	0,12	12	140	0,14
13	134	-	13	216	0,02	13	200	0,02	13	216	0,02	13	122	0,10	13	128	0,12
			14	216	-	14	200	-	14	214	0,02	14	114	0,08	14	118	0,10
									15	214	-	15	108	0,06	15	110	0,08
												16	104	0,04	16	104	0,06
												17	102	0,02	17	100	0,04
												18	102	-	18	98	0,02
												19	102	-	19	98	-
															20	98	-

When 15 ml of distilled water was added to 10 ml of pumpkin extract solution, 20 ml of 0,0001 N titrant solution was used for potentiometric titration. Taking into account that the volume of titrant consumed at the equivalent point is 5 ml, the amount of iodine in the solution was determined to be

$$\chi = \frac{N_{Na_2S_2O_3} V_{Na_2S_2O_3} Э_{KJO_3} V_{er}}{1000\ V_{ek}} = \frac{0{,}0001*20*214*10}{1000*5} = \mathbf{0{,}00085}$$ mg = **0,85** mkg was obtained.

16 ml of titrant ($Na_2S_2O_3$) was used to determine the iodine content by iodometric titration by taking 10 ml of a solution obtained by burning a mixture of palm fruit extract and potassium carbonate for analysis. The volume of titrant consumed at the equivalence point was 6 ml. Based on these data, the results of the analysis were calculated using the following formula.

$$\chi = \frac{N_t * V_t * E_a * m}{1000 \ V_{ek}} = \frac{0,01 * 16 * 214 * 10}{1000 * 6} = 0,057\ mg = 57\ mkg$$

Based on the values entered in the titration account, titration curves were constructed.

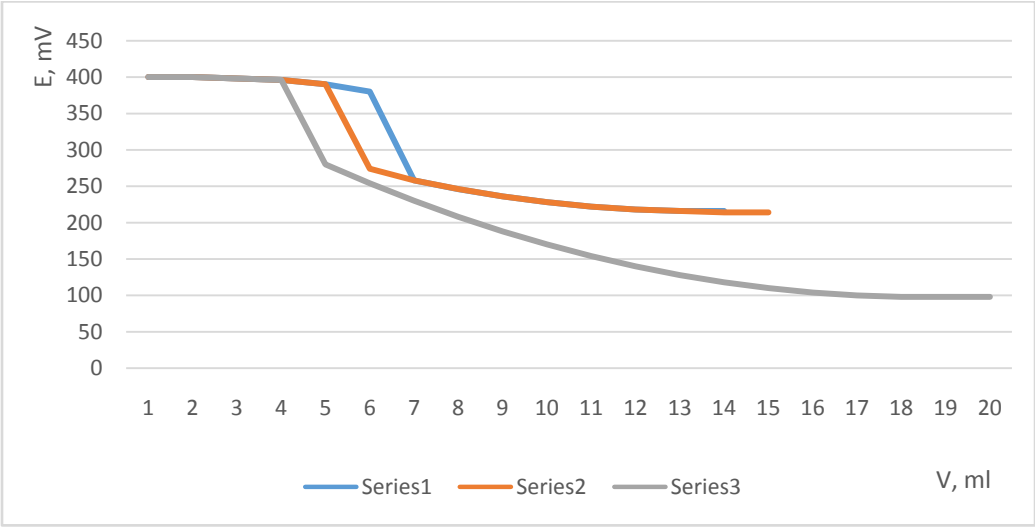

Figure 1. Graph of potentiometric titration of iodine content in persimmon, lemon peel and pumpkin fruit solutions.

When the iodine content of lemon peel extract of the same mass was determined by iodometric titration, the volume of titrant consumed at the equivalence point was 12 ml, and the total volume of titrant consumed was 28 ml. As a result of titration, the amount of iodine in the sample was found to be

$$\chi = \frac{0,0001*28*214*10}{1000*12} = 0,00049\ mg = 0,49\ mkg\ .$$

In the same order, 18 ml of titrant was used for pumpkin fruit extract. The volume of titrant consumed at the equivalence point was 5 ml. The amount of iodine as a result of titration was found to be

$$\chi = \frac{0,0001*18*214*10}{1000*5} = 0,00077\ mg = 0,77\ mkg\ .$$

The results of potentiometric determination of iodine content in solutions of persimmons, lemons and pumpkins are given in Table 2.

TABLE 2 RESULTS OF POTENTIOMETRIC AND IODOMETRIC DETERMINATION OF IODINE IONS IN PERSIMMON, LEMON PEEL AND PUMPKIN FRUIT SOLUTIONS

№	Objects to be analyzed	Theoretical amount *mkg*	*The results of the analysis, relative to 100 g of sample (χ, mkg)*	
			Potentiometric titration	*Iodometric titration*
1	***Persimmon***	60	59,9	57
2	***Lemon***	0,6	0,53	0,49

3	***Pumpkin***	1,0	0,85	0,77

If we compare the results of potentiometric and iodometric titration, we can see that the results of potentiometric titration are closer to the theoretical data.

CONCLUSIONS

The following conclusions were drawn from the experiments to determine the content of iodine in the aqueous extracts of persimmons, lemon peel and pumpkin fruits obtained as the object of study and the analysis of the results obtained in them:

1. Diospyros kaki (PCh) persimmon chocolate, Diospyros kaki (KH) Korolyok-Hyakume persimmon species, Cucurbita pepo L - pumpkin fruits and Exocarpium Citri L-lemon peel sample grown in the Andijan region as the object of analysis for the research work.

2. Aqueous extracts of the obtained samples were isolated, and analytical solutions at concentrations of 10%, 25%, 50%, 75% and 100% were prepared using bidistillate water from the initially concentrated solutions to be tested, and analytical methods were selected to study the chemical composition of these solutions.

3. Potentiometric titration was performed to determine the amount of iodine in the extracts of persimmon, pumpkin and lemon peel. The iodometric titration method was used to assess the level of accuracy of the results obtained in potentiometric titration.

4. When the content of iodine in persimmon, lemon peel and pumpkin fruits was analyzed by potentiometric and iodometric titration methods, it was found that iodine exists 59,9 mkg in 100 g in the content of persimmon, 0,53 mkg in lemon peel, 0,85 mkg in pumpkin.

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