Kinetic study of leaching behavior of electrolytic elements from leafy vegetables

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Abstract

This study aims to compare leaching of ions such as sodium, potassium, calcium under variable conditions in different leafy vegetables and suggest suitable leaching parameters. Four leafy vegetables namely, Spinach, Fenugreek, Slender Amaranth and Red calico Amaranth were selected for these studies. Three leaching conditions were carefully chosen at two different temperatures with stirring and one condition without stirring. The kinetic studies on leaching rates were carried out for the same to provide evidence for extent of leaching and time required for equilibrium leaching. It was found that by the end of 120 minutes almost 75 – 95% ions were leached out and the process followed pseudo second order mechanism. The thermodynamic studies were also carried out, that indicated that leaching was a spontaneous process with positive energy of activation under all conditions.

Keywords: Leaching, potassium, sodium, calcium, kinetics, thermodynamics.

1. Introduction

The natural source of energy to human body is food rich in nutrients, minerals and energy providers including carbohydrates and proteins. The smoot functioning of human body is directly related with consumption of food ingredients in proper proportions. All the physiological processes are governed by balanced composition of these components in foodstuff[1]. The 2020 Kidney Disease Outcome Quality Initiative (KDOQI) has come up with the conclusions which indicated that more consumption of leafy vegetables and seasonal fruits are extremely important for proper management of blood pressure and reduction of acid production[2]. Minerals found in fruits and vegetables play a crucial role in maintaining a healthy diet. Nutrition experts advise that these minerals be consumed through whole fruits and vegetables rather than dietary supplements. The recommended daily allowance (RDA) for potassium (K) in adults is between 4000 and 4700 mg, which is significantly higher than the RDA for most other minerals[3]. The RDA for calcium as well sodium is 1000 to 1600 and 2200 to 2300 mg per day respectively.

Potassium is involved in electrical and cellular body function. While high potassium levels can help to control high blood pressure and may reduce the risk of stroke[5,6]. Chronic kidney disease (CDK) is a common disorder associated with higher levels of potassium leading to risk for hyperkalemia[2]. In such cases, the potassium consumption needs to be restricted that may cause health complications[7]. Renal diseases can further lead to conditions like Hypernatremia[8] and Hypercalcemia. A decline in renal function may also be associated with other diseases and risk factors, such as hypertension and diabetes[9]. Therefore, intake of minerals is to be lowered so that further complications do not occur. This can be achieved by restricting the intake of fruits and vegetables, that may further aggravate the energy loss that can be compensated only through intake of non-veg foods which is not a primary food for most of the Indians[10]. As the major population is vegetarian and so this option is not suitable for most of the patient. So, there has to be ways to reduce the ion content from the vegetables available, so that it can preserve the diversity to the diet making the fruits and vegetables appropriate for the dietic organization of the renal diseases.

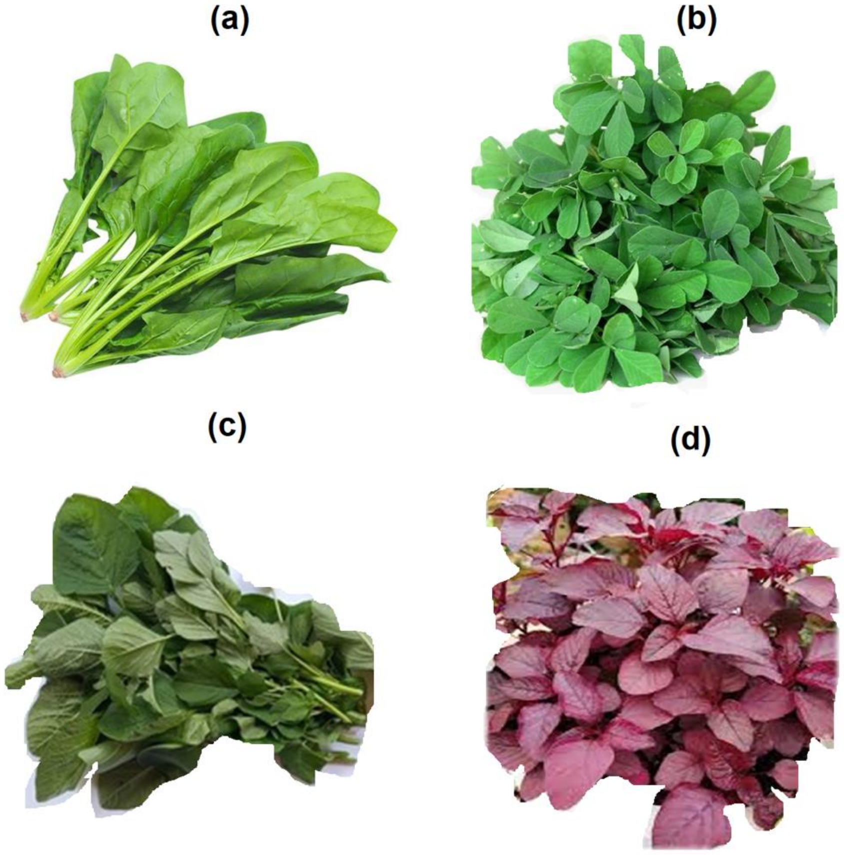
Due to complexity of lifestyle, the renal problems are increasing day by day. The reduced efficiency of kidney leading to imbalance in mineral load need to be counter by removal of excess metal ions from food materials including vegetables. Among all metal ions, potassium reduction in foodstuff is a common suggestion by dietatians. One strategy recognized to reduce potassium concentrations in foods is the use of 'wet cooking methods,' which facilitate the loss of minerals into the cooking water[11]. Several studies have investigated mineral reduction through leaching method (wet cooking method) and several different foods have been studied[3,12,13]. Also, during an opinionnaire in Chennai city in India, 65% of dieticians agreed that leaching of vegetables[14].

Leaching of metal ions has been reported by Apua and Madiba[15] for leaching of heavy metal ions from ore sample, that has led to interesting observations. But there are still many caveats in mind, that ideal strategy to employ easily and still the food remains pleasant, therefore this study aims to determine the impact of leaching the vegetables in water using different parameters. To the best of our knowledge, this is the first ever systematic study of kinetics of leaching of metal ions from leafy vegetables.

2. Materials and Methods

2.1 Reagents and leafy vegetables

Sodium chloride (NaCl), Potassium chloride (KCl), Calcium chloride ($CaCl_2$) were procured from Loba Chemie Private Limited, India, while acids such as 70% Nitric acid, 60% Per chloric acid, 35% Hydrochloric acid were purchased from Sisco Research Laboratory, India. All the chemicals were used without further purification. All the solutions were prepared in double distilled water. The four leafy vegetables spinach (*Spinacia oleracea L.*), fenugreek (*Trigonella foenum-graecum*), slender amaranth (*Amaranthus viridis*) and red amaranth (*Amaranthus cruentus*) (Photograph 1) were procured from local market, washed under running water several times and used further for analysis.



Photograph 1. The four leafy vegetables selected for leaching studies (a) spinach (b) fenugreek (c) slender amaranth (d) red amaranth

2.2 Instruments

The estimation of various metal ions was carried out using microprocessor based ELICO CL378 Flame photometer. The estimation was carried out using filters provided for each ion present. In order to maintain the temperature of the solution, MAC-Thermostat MSW-313 with temperature sensor probe and magnetic stirring facility was used. The other instruments involved were Shimadzu ATX-124 analytical balance and Equiptronics EQ-770 magnetic stirrers.

2.3 Procedure

A cascade of procedure was followed, using different parameters and conditions which included:

1. Leaching at 25^{o}C under magnetic stirring.
2. Leaching at 50^{o}C under magnetic stirring.
3. Leaching without agitation (without stirring).

Green leafy vegetables were obtained from the local market. The excessive stem and leaves were cut off and the leaves were thoroughly washed with distilled water 2-3 times in order to remove any adsorbed or deposited particles. These were then air dried for 24 h on a filter paper. After drying, these were finely minced and about 5 g weighed leaves were transferred to a 250 mL beaker. 100 mL double distilled water was added to it and the beaker was kept on a magnetic stirrer. Temperature was recorded and maintained constant throughout the experiment. Stirring was continued for 120 min. After different intervals of time 1mL of leached solution was withdrawn with the help of a micropipette from the bottom of the flask and transferred to a 10 mL of volumetric flask. Volume was made up to the mark with distilled water. This leachate solution was analyzed by flame photometry. Similar procedure was carried out for leaching at 50^{o}C.

In another study at room temperature, the system was allowed to stand still without stirring during the leaching process i.e. without agitation (WA), and the leached sample was withdrawn from bottom of the beaker to avoid any error arising due to concentration gradient developed in the system.

A series of standards prepared covering a wide range of concentration from 2 ppm to 100 ppm were aspired into the flame which was followed by the samples to estimate the ions leached from leafy vegetables. All of these studies were carried out in triplicate and the mean of three values were reported to assure high degree of precision and overcome any random errors.

For the total ion content contained in leafy vegetable sample, wet digestion method was adopted. The air-dried leaves were weighed and about 2 g of the leaves were oven dried for 48 h at 75^{o}C to ensure the complete moisture loss. The dried leaves were crushed to powder using pestle and mortar. The crushed powder was quantitatively added to a beaker, followed by 5 mL 70% nitric acid and 1.5 mL 60% perchloric acid. The solution was heated until the disappearance of brown fumes (estimated time 5 to 6 min), the solution was then cooled and to the same solution 5 mL 1:1 hydrochloric acid was added, the solution was transferred to a 25 mL volumetric flask and volume was made up to mark using double distilled water. This was analyzed by flame photometry. In order to get analytical precision, all the experiments were carried out in triplicate and mean values were reported in all the cases.

2.4 Kinetics models

The leaching of metal ion by the leaves depends on the contact time, and in this context the study of the kinetics of leaching is of considerable significance. The pseudo-first-order and pseudo-second-order kinetic models were utilized to fit the experimental data with the theoretically derived data.

The pseudo-first-order kinetics[16] is given by-

$$\log{(q_e - q_t)} = log\ q_e - \frac{k_1 t}{2.303} \text{- - - - - - - -} (1)$$

where q_e and q_t refers to the amounts of ions leached (mg g^{-1}) at equilibrium and at time t, respectively, with the first-order rate constant k_1. The plot of log (q_e - q_t) against t gives the pseudo-first-order rate constant (min^{-1}).

The pseudo-second-order[17] equation is given as-

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{t}{q_e} \text{- - - - - - - -} (2)$$

where k_2 is the pseudo-second-order rate constant (g mg^{-1} min^{-1})

The values of correlation coefficient were calculated for both the orders and value of correlation coefficient which was found to be close to 1 leaching was considered to follow that particular order. From the values obtained for both models, rate constants were calculated at two different temperatures, which in turn were used to obtain the values of activation energy using Arrhenius equation –

$$ln \frac{k\prime}{k} = \frac{E_a}{R}\left(\frac{T_2 - T_1}{T_1 T_2}\right) \text{- - - - - - - -} (3)$$

Where, k and k’ are the rate constants of best fit model at temperatures T_1 and T_2 (K) respectively, E_a (kJ mol^{-1}) is the activation energy and R (kJ mol^{-1} K^{-1}) is the universal gas constant.

Another theoretical model that is commonly used mainly for adsorption studies is Weber-Morris intraparticle diffusion model[18]. It is expressed by equation (4)

$$q_t(mg/g) = k_{int}t^{1/2} + c\text{- - - - - - - -}(4)$$

Where k_{int} is the intraparticle diffusion constant and c represents thickness of layer. The values of k_{int} and c were obtained by plotting a graph of q as a function of $t^{1/2}$. The process is diffusion controlled if the intercept c is zero.

3. Results and discussion

3.1. Batch leaching studies

The concentration of leachates under three experimental conditions was determined by pipetting 1 mL of the leachate at different time intervals from the bottom of the beaker. The values of concentration of potassium, sodium and calcium in leachates were found to increase with time in all the four vegetables. Representative variation graphs of potassium have been shown in Fig. 1. The other two metal ions followed the same trend.

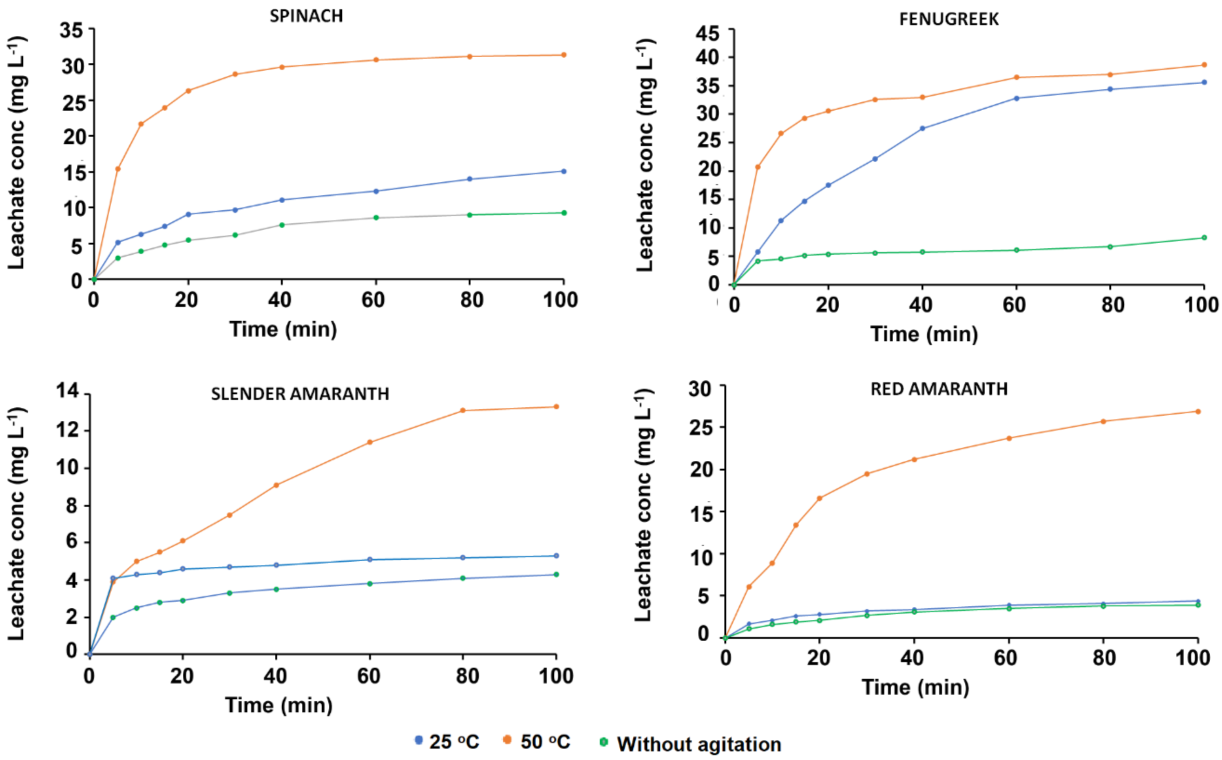


Fig. 1. Variation of leachate potassium concentration with time under three experimental conditions for the four vegetables

It was observed that the rate of leaching was maximum under stirring condition at 50 ^{o}C while it was minimum in non-stirring condition at room temperature. It was observed that the agitation as well as elevation in temperature lead to rapid establishment of equilibrium as well as higher degree of leaching. This observation was in agreement with the results reported for lead leaching from galena ore[19] or potassium from spinach or escarole[20].

In order to establish the extent of leaching, the maximum degree of leaching of each ion from each vegetable was compared with the total metal content of that vegetable, as estimated by wet digestion method. The total metal concentration and the amount leached at 50 ^{o}C under stirring condition have been presented in Table 1. It can be interpreted from this table that about 90% of these metal ions get leach out at equilibrium at elevated temperature of 50 ^{o}C from all the four vegetables. It means that, the patient who is suffering from renal problems and advised to reduce the metal ion intake may get rid of these ions by soaking them in hot water resulting in substantial reduction in their concentration in vegetables.

Table 1. Total metal content versus leaching at equilibrium at 50 ^{o}C

Vegetable	Sodium			Potassium			Calcium		
	Total (mg/g)	Leached at equilibrium (mg/g)	Leaching percentage (%)	Total (mg/g)	Leached at equilibrium (mg/g)	Leaching percentage (%)	Total (mg/g)	Leached at equilibrium (mg/g)	Leaching percentage (%)
Spinach	9.91± 0.82	9.90± 0.42	99.8	6.72± 0.89	6.31± 0.33	93.8	93.62± 1.51	81.4± 1.66	86.9
Fenugreek	3.00± 0.45	2.18± 0.22	72.6	12.06± 1.72	7.78± 1.88	64.5	79.76± 3.22	78.25± 3.83	98.1
Slender Amaranth	0.76± 0.13	0.74± 0.21	97.3	2.78± 0.73	2.70± 0.64	97.1	14.25± 0.25	13.86± 2.43	97.2
Red Amaranth	1.83± 0.30	1.42± 0.03	77.5	5.64± 0.99	5.48± 0.33	97.1	36.55± 4.02	34.62± 2.76	94.7

3.2 Kinetics of leaching

The three kinetics models, namely pseudo first order, pseudo second order and intraparticle diffusion were applied to leaching process. It has been observed that the pseudo second order kinetics model fits well for all leaching of all the three ions in each vegetable under all the three experimental conditions. This has been concluded based on the fact that the coefficient of determination (R^2) is very close to 1 in this kinetics model. This shows that the leaching rate is controlled by the sample dissolution after complete suspension in water[21]. Representative graphs of potassium leaching have been shown in Fig. 2 while similar trend was observed for other metal ions.

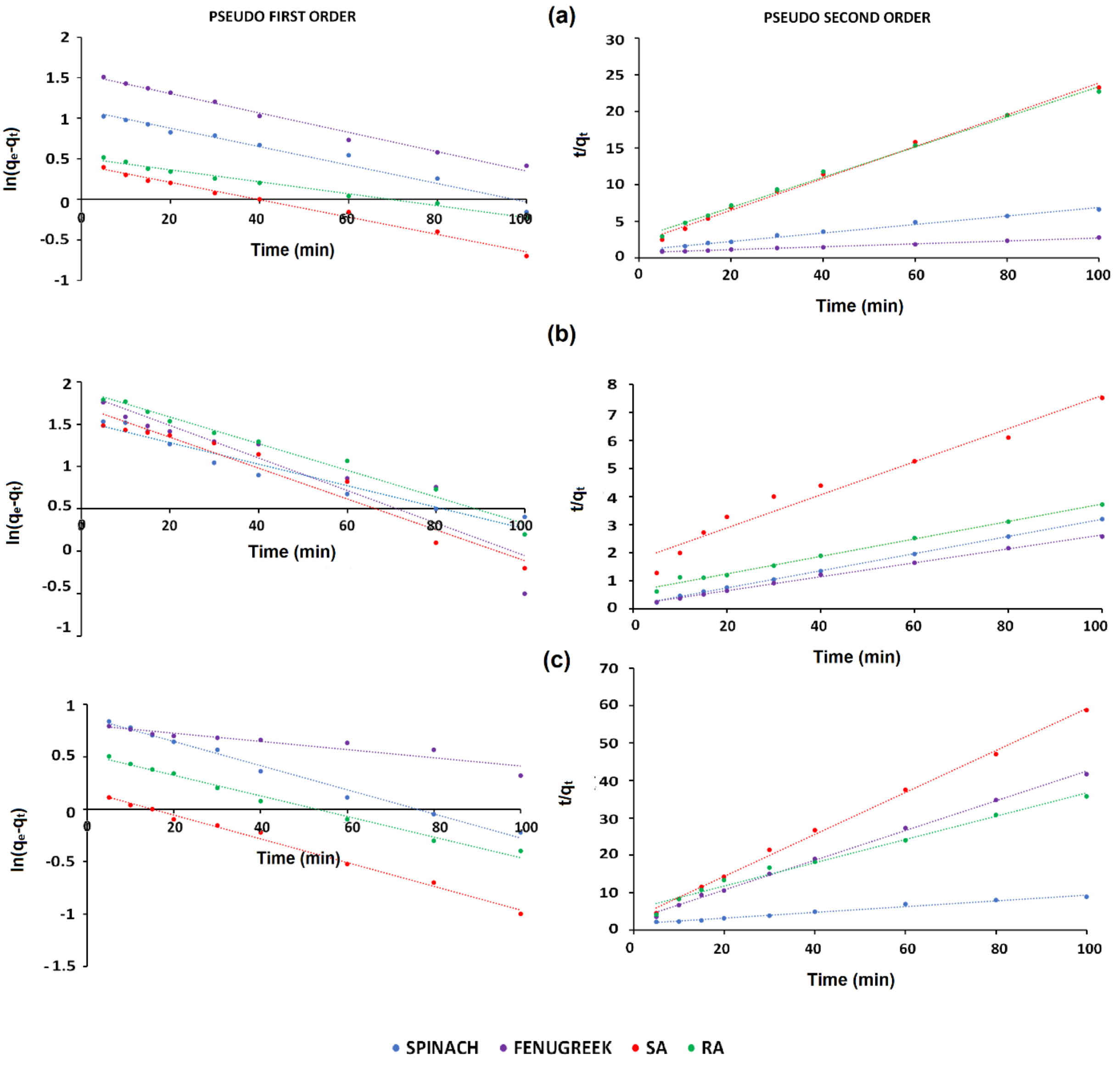


Fig. 2. Pseudo-first-order and pseudo-second-order kinetics models for leaching of potassium under three experimental conditions: (a) 25 ºC with agitation (b) 50 ºC with agitation (c) 25 ºC without agitation.

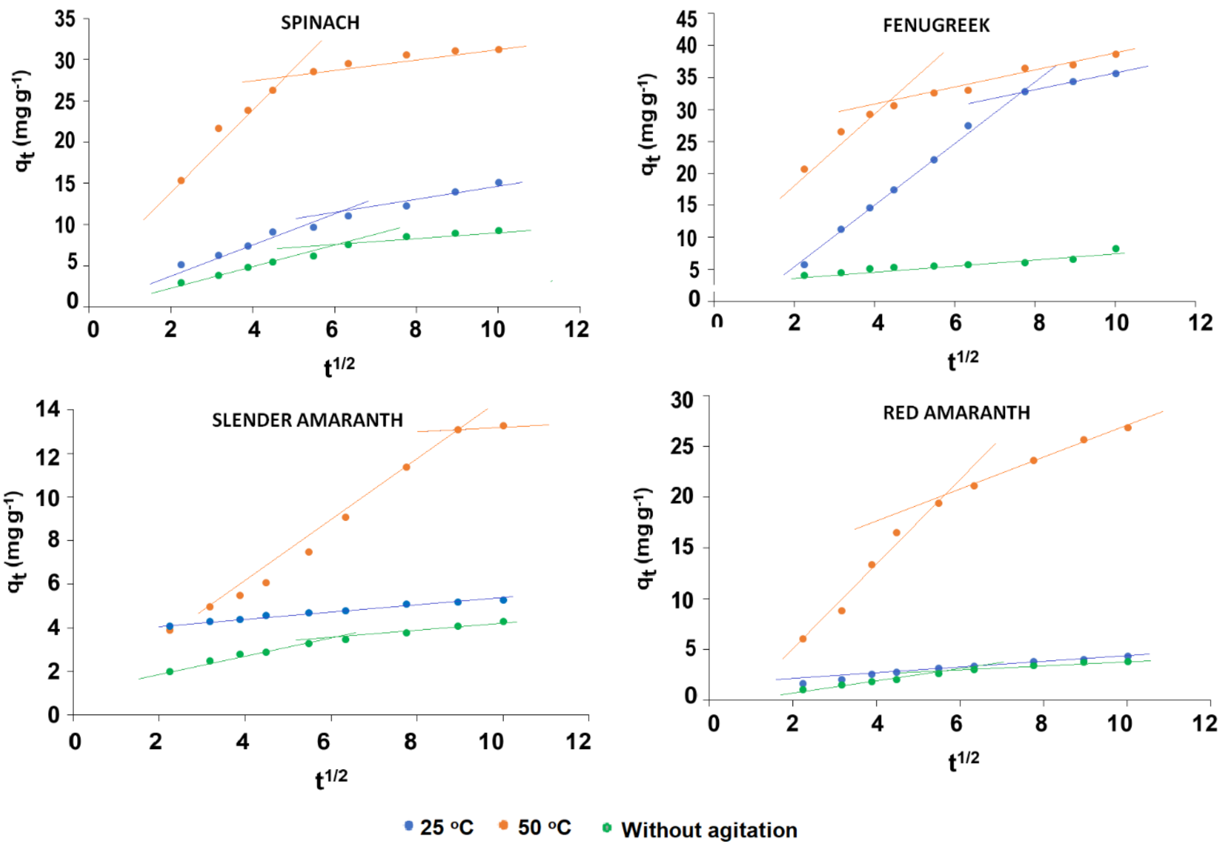


Fig. 3. Intraparticle diffusion model for leaching of potassium under three experimental conditions.

Table 2. Kinetics studies of metals leaching process

Vegetable	**Pseudo-first-order**					
	25 °C		50 °C		Without agitation	
	K_1 (min^{-1})	R^2	K_1 (min^{-1})	R^2	K_1 (min^{-1})	R^2
			Potassium			
Spinach	0.0264	0.9886	0.0277	0.9216	0.0214	0.9890
Fenugreek	0.0275	0.9875	0.0285	0.9756	0.0092	0.8702
SA	0.0085	0.7249	0.0269	0.9697	0.0016	0.4364
RA	0.0126	0.8967	0.0298	0.9886	0.0124	0.8291
			Sodium			
Spinach	0.0149	0.8947	0.0201	0.9566	0.0077	0.9519
Fenugreek	0.0207	0.9675	0.0237	0.9175	0.0099	0.9224
SA	0.0260	0.9923	0.0306	0.9106	0.0097	0.9425
RA	0.0290	0.8634	0.0337	0.9703	0.0203	0.9757
			Calcium			

Spinach	0.0235	0.9464	0.0346	0.9578	0.0210	0.9640
Fenugreek	0.0359	0.9898	0.0382	0.8596	0.0122	0.9084
SA	0.0179	0.9502	0.0306	0.9817	0.0174	0.9792
RA	0.0270	0.9502	0.0478	0.8199	0.0189	0.9286
	Pseudo-second-order					
	K_2 ($L\ mg^{-1}\ min^{-1}$)	R^2	K_2 ($L\ mg^{-1}\ min^{-1}$)	R^2	K_2 ($L\ mg^{-1}\ min^{-1}$)	R^2
			Potassium			
Spinach	0.0548	0.9849	0.2270	0.9994	0.0543	0.9938
Fenugreek	0.0950	0.9934	0.1626	0.9981	0.0286	0.9593
SA	0.0993	0.9950	0.2991	0.9604	0.0346	0.9989
RA	0.0493	0.9944	0.9738	0.9925	0.0481	0.9950
			Sodium			
Spinach	0.1371	0.9995	0.3617	0.9946	0.0463	0.9842
Fenugreek	0.1439	0.9782	0.1844	0.9959	0.0326	0.9976
SA	0.0918	0.9931	0.1787	0.9526	0.0381	0.9974
RA	0.0563	0.9977	0.1492	0.9716	0.0287	0.9820
			Calcium			
Spinach	0.1159	0.9900	0.2059	0.9991	0.0871	0.9864
Fenugreek	0.0738	0.9990	0.1766	0.9971	0.0693	0.9870
SA	0.0780	0.9903	0.1047	0.9882	0.0427	0.9879
RA	0.0536	0.9968	0.1889	0.9835	0.0253	0.9791
	Intraparticle diffusion					
	K_{int}	c	K_{int}	c	K_{int}	c
			Potassium			
Spinach	1.2708	2.6465	1.7781	16.179	0.8502	1.4984
Fenugreek	3.9757	0.6526	2.0155	19.968	0.4317	3.2609
SA	0.2821	1.6069	1.3286	0.6108	0.1553	3.8207
RA	0.3391	1.1648	2.6573	2.5766	0.3732	0.4671
			Sodium			
Spinach	0.2828	7.3918	2.7170	20.341	1.0156	1.3273
Fenugreek	0.4224	0.0925	0.4608	5.8475	0.1307	1.1746
SA	0.1553	0.8207	0.3269	0.2799	0.0780	0.9805
RA	0.0668	0.5230	0.7628	0.0944	0.2318	0.5656
			Calcium			
Spinach	2.2435	17.722	14.1739	173.6686	1.4417	8.4543
Fenugreek	4.0947	11.4899	4.1800	49.7948	10.5641	30.1083
SA	1.8518	7.0852	6.4317	6.5925	2.1509	16.9493
RA	1.6816	22.1950	18.8006	6.2842	2.0428	4.5323

As the intraparticle diffusion model shows two-step process before reaching the equilibrium (Fig. 3). The initial linear increase in q value represents external surface desorption while the second step is mainly diffusion dependent. As the agitation led to enhancement in leaching rate and rapid establishment of equilibrium, it indicated that the process was diffusion controlled in the beginning. However, the non-zero intercept (c) in all the cases indicate that the overall process is not controlled by diffusion phenomenon, even in condition where there is no agitation or stirring[22].

The high values of activation energies ranging from 7.95 to 95.45 kJ mol^{-1} (Table 3) indicate that the rate of leaching is highly influenced by temperature and is it a surface-controlled reaction[22]. The lower values of activation energy are indicative that the effect of solution temperature is less while the higher activation energy indicate higher impact of temperature on leaching process.

Table 3. Energy of activation for leaching process

Vegetable	E_a (kJ mol^{-1}) for leaching of		
	Potassium	**Sodium**	**Calcium**
Spinach	45.47	31.04	18.39
Fenugreek	17.20	7.95	27.90
SA	35.29	21.31	9.41
RA	95.45	31.18	40.29

3.3 Comparison with reported literature

There are hardly any studies reported on the kinetics of the leaching process from leafy vegetables in the literature. However, a few workers have reported the leaching percentage of potassium ions. Table 4 depicts the comparison of the results obtained in these studies with those reported in literature. The literature shows lower leaching even at higher temperature as the studies have been carried out for just 5 min.[20] Another report [23] shows that increasing the leaching time to 240 min at room temperature also does not lead to effective leaching. Our studies have systematically reported maximum leaching under moderate temperature that too in intermediate time span.

Table 4. Comparison with leaching percentage reported in literature

Vegetable	Temperature (°C)	Time (min)	Metal ion leaching (%)			Reference
			Sodium	Potassium	Calcium	
Spinach	50	100	99.8	93.8	86.9	This work
Fenugreek	50	100	72.6	64.5	98.1	This work
SA	50	100	97.3	97.1	97.2	This work
RA	50	100	77.5	97.1	94.7	This work
Spinach	100	5	-	28	-	Abreu et al [20]
Escarole	100	5	-	56	-	Abreu et al [20]
Amaranth leaves	25	240	-	36.4	-	Srilakshmi et al [23]
Agathi leaves	25	240	-	34.5	-	Srilakshmi et al [23]

4. Conclusion

To prevent complications such as cardiac arrhythmias and sudden death, it is important to control potassium intake in the nutritional management of chronic kidney disease. The highest percentage of leaching was observed in the case of higher temperature (50°C) thus temperature can affect the kinetics of leaching which concluded that increase in temperature helps to leach out higher percentage of ions from leaves. And by the end of 120 minutes almost all ions leached out from the samples completely. Numerous studies have examined the impact of mixing conditions on the leaching rate, finding that the rate initially increases before leveling off as the mixing speed continues to rise. It was also found that, as the correlation coefficient value for pseudo-second-order was close to 1 in all the cases, it was therefore concluded that leaching of ions from vegetables strictly followed the particular order meaning the rate of reaction depend upon the concentration of both the vegetables as well as the water content in which they were being leached out.

It is a general observation that most biological samples of plant origin and especially leafy vegetables are rich in K compared to Na as can be seen in Table 1 where except spinach all other 3 leafy vegetables show higher K content. Also, it is interesting to note that all three electrolytic elements are leached out in hot water to the extent of >90%. Undoubtedly and obviously, leaching is maximum at 50 °C in all cases. Most commonly in Indian kitchen, these vegetables are cooked in water which is not thrown away. Therefore, an obvious conclusion may be that if one does not want to consume K then leachate water should be thrown away.

Authors Contributions

PT: Formal analysis, Methodology, Writing Original draft.

RJ: Conceptualization, validation, Resources, Writing-Finalization.

Conflict of Interest

The authors declare no conflict of interest in this work.

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