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Comparison of potentiometric, pH, and indicator titration methods for the detection of ibuprofen in tablets and suspensions

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Abstract
Ibuprofen has anti-inflammatory effects. It is a weak monobasic organic acid. It is insoluble in water but can be dissolved in organic solvents such as methanol and ethanol. This research aimed to determine the ibuprofen content in two pharmaceutical forms using potentiometric, pH, and indicator titration methods: compressed tablets and suspensions. Ibuprofen tablets and suspensions were purchased from various pharmacies in Ibb, Yemen. The titrations were conducted using a sodium hydroxide solution as the titrant. The three methods had no significant differences, but the potentiometric and pH methods were the most effective for estimating ibuprofen in colored suspensions. Additionally, the study revealed no significant difference between the measured ibuprofen content and the amount stated on the product label in Ibb city, Yemen.

Keywords: Indicator, potentiometric, ibuprofen, pH, titration, suspension

1. Introduction
Ibuprofen is a weak organic acid with a molecular weight of 206.4 g/mol and is an anti-inflammatory drug class (NSAID) that primarily medicates headaches, back pain, etc. [1, 2]. It exists in pharmacies in various forms such as capsules, tablets, suspensions, and syrups which, are frequently used compared to other drugs such as Aspirin, diclofenac sodium, and pyrazolone classes [3]. It contains a carboxylic group, making it a monobasic molecule with a molar ratio of 1:1 as illustrated in Fig. 1, and it reacts with a strong base like sodium hydroxide solution.
To detect any active pharmaceutical ingredients (APIs) based on the chemical composition of drug molecules, factors such as solubility, stability, pH, excipients, and usage form must be considered [4]. The ibuprofen content was usually determined by different analytical techniques, such as ultraviolet-visible (UV-VIS) spectroscopy [5-7], high – performance liquid chromatography (HPLC), and titrations [8].
Pharmaceutical instrumental analysis approaches require advanced tools that are economically demanding. In contrast, titration methods such as visual, pH, and potentiometric titrations can be used for measuring ibuprofen content with a strong base such as sodium hydroxide solution. These methods are widely used to detect active ingredients in laboratories due to their ease of use and low instrument cost. However, these methods are not suitable for pharmaceutical products with other acidic compounds; therefore, we advise isolation before detection [9].
Indicator titration involves an observable color change in the indicator, such as a change in ph. ph. However, potentiometric and pH titrations depend on the consumed volume of the titrant through the potential difference between two electrodes in the potentiometric curve [10].
In the present research, the ibuprofen content in suspensions and tablets is a widely used NSAID class and is accessible over the counter (OTC) in most countries [11] was quantified by an indicator, pH, and potentiometric titrations, and a comparison was made. The three methods have been examined to determine an easy strategy to measure the ibuprofen content in two pharmaceutical forms in Ibb, Yemen.

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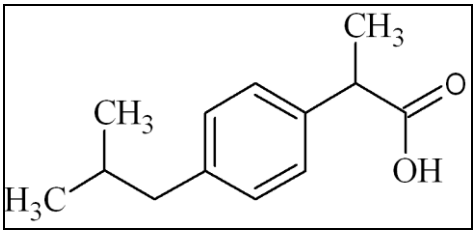


Fig 1: Chemical structure of ibuprofen

2. Materials and Methods

2.1 Preparation of instruments and chemicals

All glassware and chemical substances, including 0.100 M potassium hydrogen phthalate (KHP), 96% ethanol (v/v), a buffer solution at pH 4 and 10, and a calibrated pH meter, were used. A phenolphthalein solution at 2.00% (w/v) was used as an indicator for the analysis. The pH electrode (PHS -3E pH meter) was washed well with di-ionized water and then calibrated with a buffer solution. Then, the pH electrode was again mixed with di-ionized water.

2.2 Preparation of reagents

Approximately 1.000 g and 4.100 g of dried sodium hydroxide pellets were carefully transferred into cleans volumetric flasks and dissolved in 100 ml of deionized water. Each solution was then filled until it reached the calibrated line 250 ml. These solutions were standardized using a 0.100 M potassium hydrogen phthalate solution. The molarity of the sodium hydroxide solutions was standardized at 0.095 M and 0.400 M.

2.3 Sample collection

Ibuprofen samples in Table 1 were purchased from various pharmacies in Ibb, Yemen, which are considered suitable for pharmacies available in Yemen with different batch numbers. The selected samples included five brands of 100 mg/5 ml suspensions and five brands of 400 mg/table ibuprofen tablets.

2.4 Preparation of a standard, isolation, and determination of a pure ibuprofen (IBU) sample

Two tablets of ibuprofen were crushed into a fine powder, dissolved in 20 ml of deionized water, and stirred for 15 minutes. The ibuprofen solution was mixed with 20 mL of chloroform. The organic layer was removed. Then, 20 ml of chloroform was poured into an aqueous solution. A needle-like crystal was obtained after filtration and solvent evaporation. A weight of 1.00 g was obtained, and a melting point analysis was, then performed to ensure that the powder was extracted. The melting point of the extracted sample was 75–77 °C. The sample was weighed and detected using a 0.095 M sodium hydroxide solution and titrated until it reached a pink color, using a ph. ph as an indicator.

2.5 Sample preparation

The compressed tablet samples were prepared using a mortar to cursh into a fine powder well. The samples were mixed well before use and were maintained at 25°C while suspension samples were prepared using 10 ml of the original suspension after shaking the bottle well. The suspension sample was diluted using 10-20 ml of ethanol, stored in a container at a temperature not exceeding 25 °C and protected from sunlight.

2.6 Determination of compressed ibuprofen tablets using the indicator titration method

The powder sample was placed into a cleansing conical flask and then dissolved in 30 ml of ethanol and 20 ml of di-ionized water. Then, as an indicator, 0.4 ml of phenolphthalein solution was added. The solution was gradually titrated with a 0.095 M sodium hydroxide solution until a pink color was obtained. The volume of the base consumed was recorded as two significant figures after the decimal point, and this process was carefully repeated five times. This procedure was followed for all brands.

2.7 Determination of ibuprofen suspension using the indicator titration method

The prepared solution was gradually titrated with 0.095 M sodium hydroxide solution until a pink color was obtained after adding 0.4 ml of phenolphthalein solution. The volume of the base consumed was recorded as two significant figures after the decimal point, and this process was carefully repeated five times. This procedure was followed for all brands.

2.8 Determination of compressed ibuprofen tablet using pH and potentiometric titration methods

The represented fine powder of ibuprofen was dissolved in 10 ml of ethanol and 10 ml of di-ionized water. The pH and potential values were measured before adding any titrants. The solution was titrated using a 0.095 M sodium hydroxide solution in a potentiometric titration. Additionally, it was titrated using a 0.400 M sodium hydroxide solution while recording pH values, and both pH and potential were recorded again. The sodium hydroxide solution was added incrementally, and the pH and potential were noted after each addition. This process continued until the desired data were obtained in Tables No. 4 and 5. This procedure was followed for all brands. The data were graphed in Excel, plotting the pH/potential values versus the consumed volume of sodium hydroxide solution to obtain a "zero derivative curve" or pH curve. The first and second derivative curves were created from the data.

2.9 Determination of ibuprofen suspension using pH and the potentiometric titration methods

The pH and potential were measured for a prepared solution before adding any titrant. The solution was titrated using a 0.095 M sodium hydroxide solution in a potentiometric titration. Additionally, it was titrated using a 0.400 M sodium hydroxide solution while recording pH values, and both pH and potential were recorded again. The sodium hydroxide solution was added incrementally, and the pH and potential were noted after each addition. This process continued until the desired data were obtained. This procedure was followed for all brands. The data were graphed in Excel, plotting the pH and potential values versus the consumed volume of sodium hydroxide solution to obtain a "zero derivative curve" or pH curve. The first and second derivative curves were created from the data.

2.10 Statistical analysis

Statistical analysis was conducted using one-way analysis of variance (ANOVA). The means and graphs were calculated using Microsoft Excel 2019.

3. Results and Discussion

3.1 Determination of the endpoint

The endpoint was observed using three various titration methods:

3.1.1 Indicator titration

The study used a phenolphthalein solution to monitor endpoints in all studied samples. When the standard sodium hydroxide solution was added to the ibuprofen solution, a gradual color change occurred after each addition until the light pink color stabilized, representing the endpoint in Table 2. The average amount of sodium hydroxide solution was 19.2-19.5 ml for the tablets and 4.32-4.56 ml for the suspensions. Fig. 2 illustrates the ibuprofen content was computed based on this endpoint. The ibuprofen content was identical to the quantity listed on the label, with relative errors ranging from -0.93 to 0.104 for the tablets and from -15.30 to -9.02 for the suspensions.

3.1.2 Potentiometric Titration

The potential values were recorded after adding 1.00 ml of standard sodium hydroxide solution until the endpoint was reached as illustrated in Tables 3A and 3B. The potentiometric curves demonstrated that the endpoint was determined at the intersection of the potential curve and the x-axis, ranging from 20.25 to 20.60 ml for tablets and 9.40 to 9.70 ml for suspensions as shown in Fig. 3. This method determined that the ibuprofen contents were 397.10–402.00 mg per tablet and 95.10–92.20 mg per 5 ml of the original suspensions as shown in Tables 3C and 3D these data represented in Fig. 3.

3.1.3 pH titration method

The pH values were taken after each addition, and a sharp change in pH values that represented the endpoint region was observed as shown in Figs. And Tables 4. Then, the second derivative curve was plotted to obtain a more precise and accurate endpoint. The endpoint was determined at the intersection of the second derivative curve and the x-axis, ranging from 2.5 to 2.6 ml for suspensions as seen in Figs. 4. This method determined that the ibuprofen content was 103.2–107.30 mg / 5 ml of the original suspension as shown in Tables 4E and 4F.

3.2 Determination of the ibuprofen content in compressed tablets and suspensions using titration methods

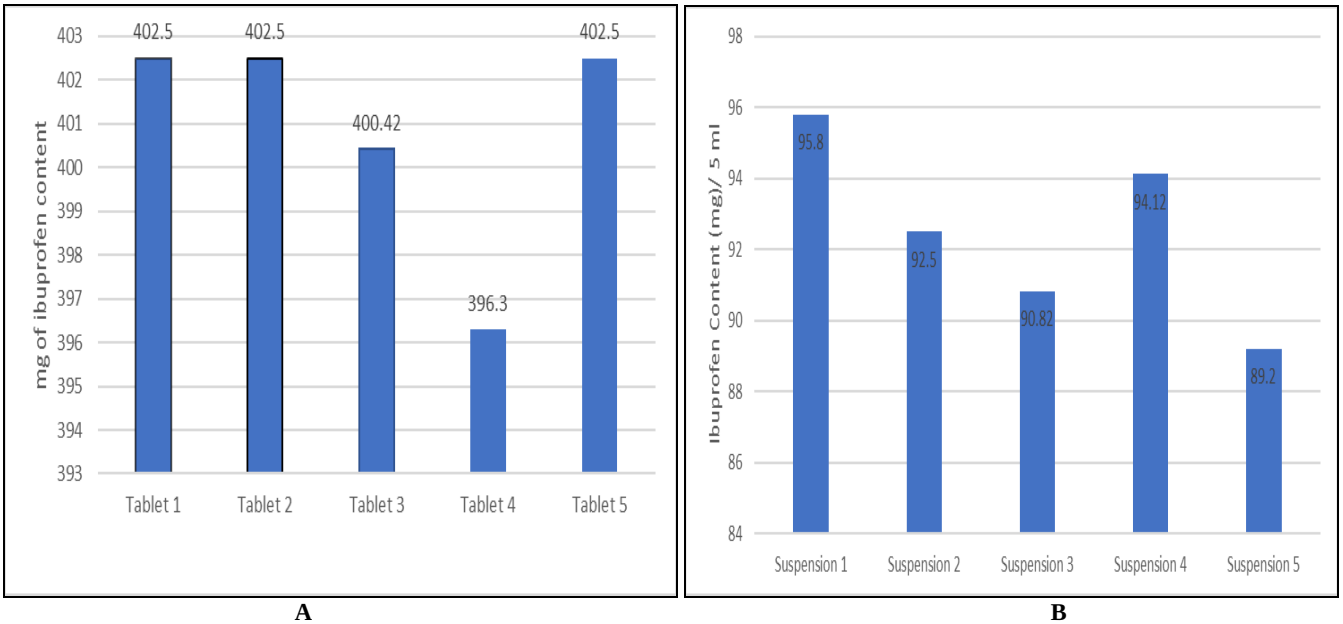
The ibuprofen content in the compressed tablets and suspensions was determined using the following equation:

mg of ibuprofen = M ✕ V ✕ M.wt. of ibuprofen

where M refers to the molarity of the sodium hydroxide solution and V represents the consumed volume of NaOH solution until it reaches the endpoint.

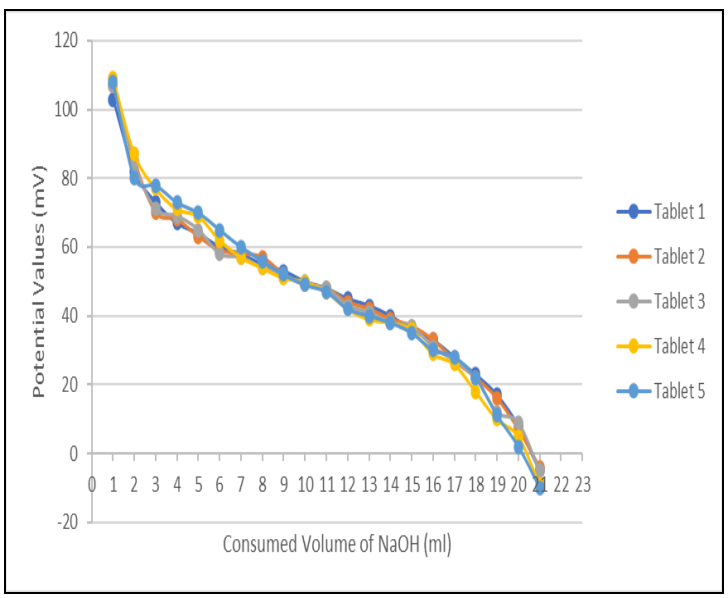
3.3 Comparative analysis of indicator, pH, and potentiometric titration methods

An evaluation of the three methods was performed, and it was noted that there were no considerable differences in the compressed tablets among all three methods used. However, for suspensions, pH and potentiometric titration methods were noted to be the most effective, as shown in Figs. 5.

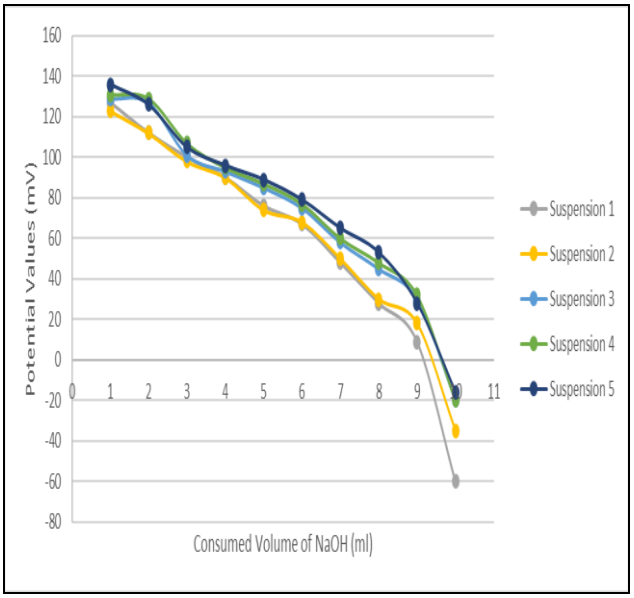


A **B**

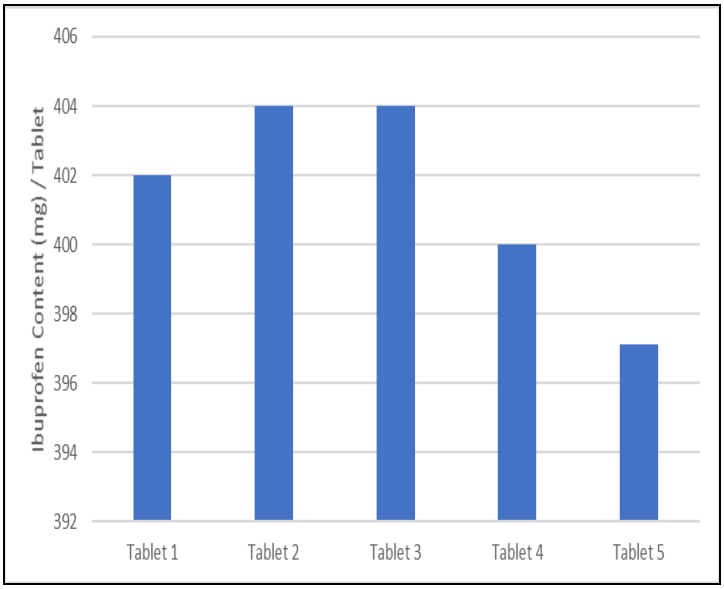
Fig 2: Amount of ibuprofen contents in tables (A) and suspensions (B) using the indicator titration method.



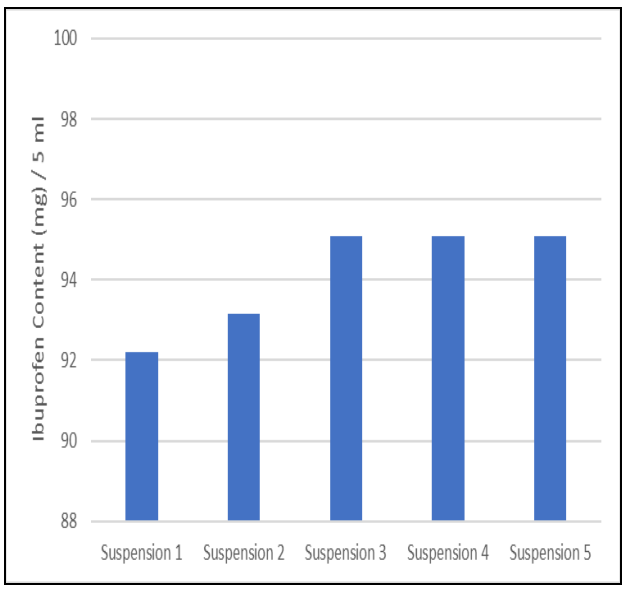
A



B

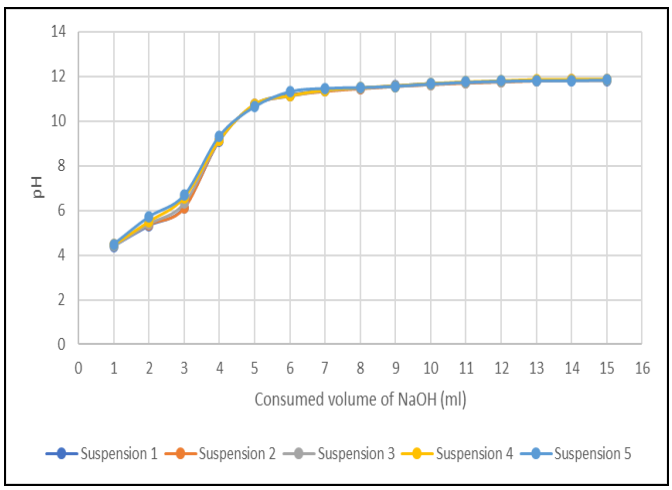


C

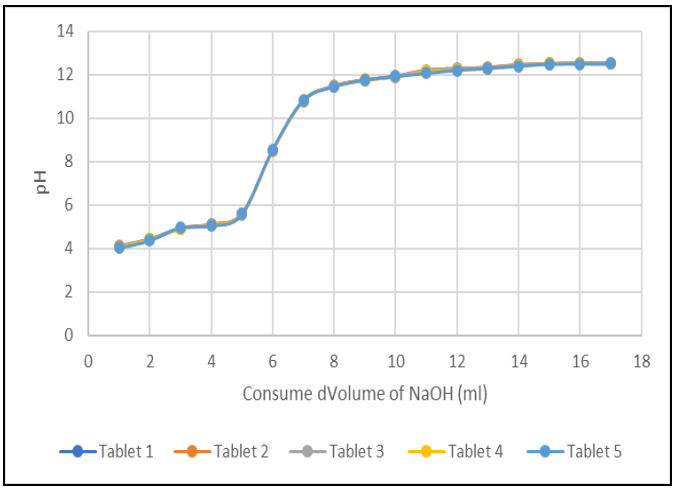


D

Fig 3: The endpoint detection using the potentiometric titration method and amount of ibuprofen contents in tablets (C) and suspensions (D) using the potentiometric titration method



A



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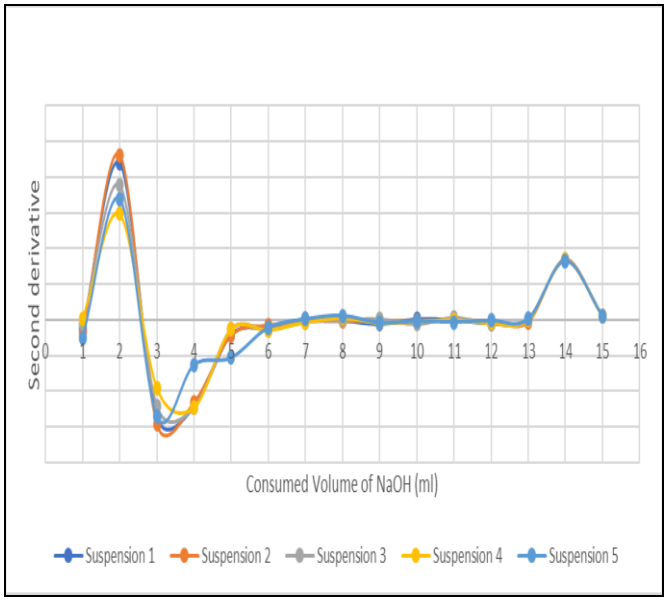
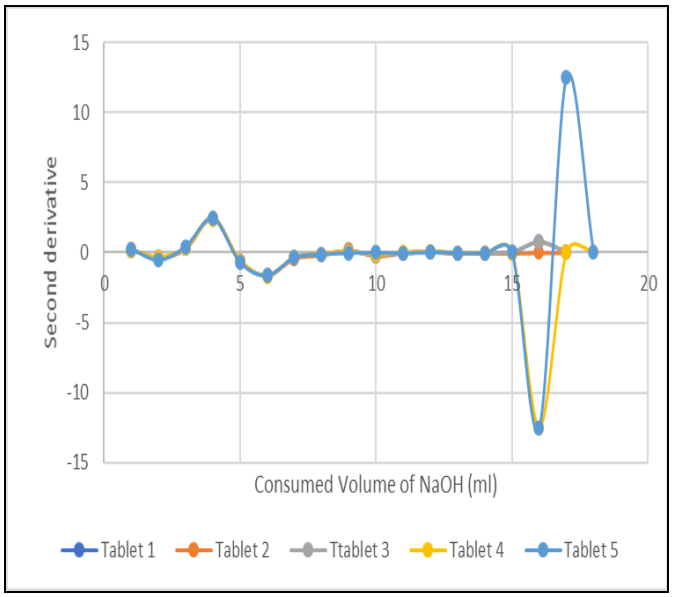
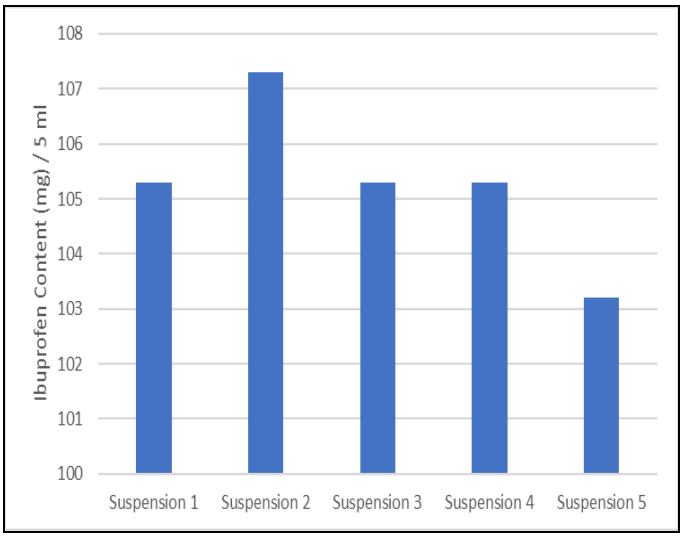


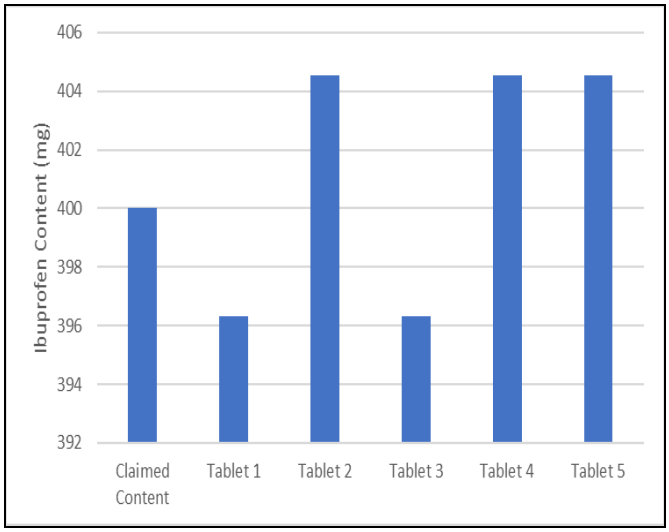
C



D

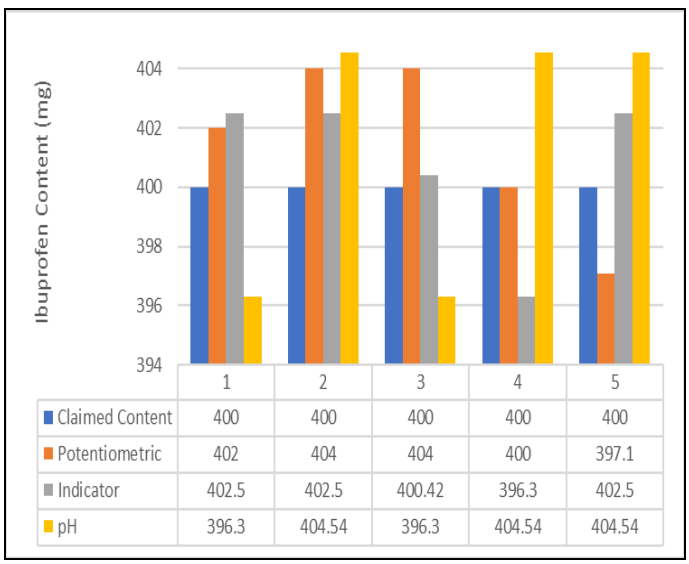


E



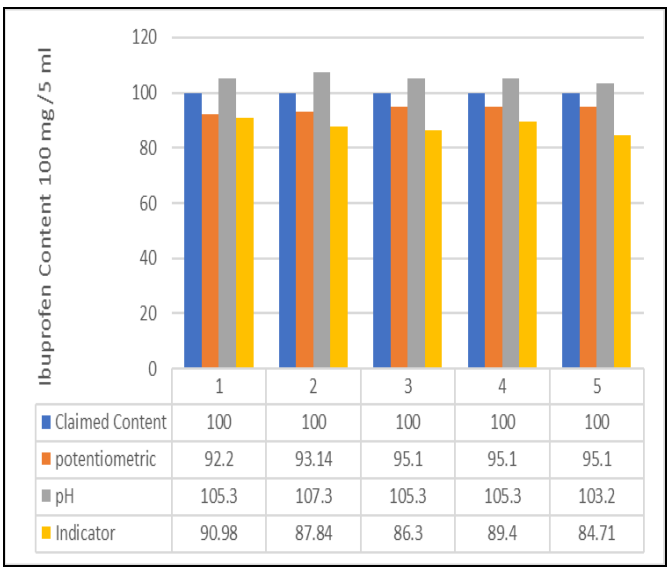
F

Fig 4: The second derivative endpoint detection suspension (C) tablets (D), pH curve (A, B for suspensions and tablets respectively), and amount of ibuprofen contents in suspensions (E) and tablets (F) using the potentiometric titration method.



	1	2	3	4	5
Claimed Content	400	400	400	400	400
Potentiometric	402	404	404	400	397.1
Indicator	402.5	402.5	400.42	396.3	402.5
pH	396.3	404.54	396.3	404.54	404.54

A



	1	2	3	4	5
Claimed Content	100	100	100	100	100
potentiometric	92.2	93.14	95.1	95.1	95.1
pH	105.3	107.3	105.3	105.3	103.2
Indicator	90.98	87.84	86.3	89.4	84.71

B

Fig 5: Comparison of the potentiometric, pH, and indicator titration methods and titrimetric methods in compressed tablets (A) and suspensions (B).

Table 1: Label information of the selected brands of ibuprofen tablets and suspensions

Code	Brands	Expiry Date (mm. yyyy)	Batch Number
Tablet 1	Ibuprofen Denk	01.2026	455 PC
Tablet 2	Ibuprofen M	11.2026	356727
Tablet 3	Ibupen	01.2027	Yu328
Tablet 4	Ibufen	07.2026	Tstd87
Tablet 5	Ibumen 400	05.2026	612673
Suspension 1	Pofen 100	01.2026	455 PC
Suspension 2	Improfen	02.2027	24091
Suspension 3	Isofen	05.2027	1426378
Suspension 4	Brufemol	12.2026	123657
Suspension 5	Dolofen	10.2025	22375

Table 2A: Ibuprofen content in compressed tablets using an indicator titration method with means and purity.

No.	Brands	The volume of titrant (ml)	Means $\pm SD$	Measured mg of IBU /tablet	Claimed label (mg)	Relative error	% Purity
01	Tablet 1	19.50 20.00 19.50 19.50 19.00	19.5 ± 0.35	402.**50**	400	0.62	100.63
02	Tablet 2	19.00 19.50 19.00 20.00 20.00	19.5 ± 0.35	402.**50**	400	0.62	100.63
03	Tablet 3	18.50 19.00 20.00 20.00 19.50	19.4 ± 0.65	400.4**2**	400	0.104	100.105
04	Tablet 4	19.00 18.50 20.00 19.50 19.00	19.2 ± 0.57	396.**30**	400	-0.93	99.07
05	Tablet 5	19.00 19.50 20.00 19.50 19.50	19.5 ± 0.35	402.**50**	400	0.62	100.63

Table 2B: Ibuprofen content in suspension using an indicator titration method with means and purity.

No.	Brands	The volume of titrant (ml)	Means $\pm SD$	Measured mg of ibuprofen/tablet	Claimed label (mg)	Relative error	% Purity
01	Suspension 1	3.8 4.2 5.2 4.8 5.2	4.6 ± 0.62	90.98	100	-9.02	95.8
02	Suspension 2	4 4.4 4.8 5.2 4	4.48 ± 0.52	87.84	100	-12.16	92.5
03	Suspension 3	4 4.4 4.8 3.6 5.2	4.4 ± 0.63	86.30	100	-13.73	90.82
04	Suspension 4	4.4 4.8 4.4 5.2 4	4.56 ± 0.46	89.40	100	-10.60	94.12
05	Suspension 5	4	$\pm$	84.71	100	-15.30	89.2

		4.8					
		4.8					
		4.4					
		3.6					

Table 3A: Result of millivolts of ibuprofen in compressed table solutions using a potentiometric titration method.

Consumed volume of NaOH (ml)	Millivolt of tablet 1	Millivolt of tablet 2	Millivolt of tablet 3	Millivolt of tablet 4	Millivolt of tablet 5
0	103	107	107	109	108
1	82	85	84	87	80
2	73	70	71	77	78
3	67	68	69	71	73
4	64	63	65	69	70
5	60	59	58	62	65
6	58	58	57	57	60
7	55	57	54	54	56
8	53	52	52	51	52
9	50	49	49	50	49
10	48	48	48	47	47
11	45	44	43	42	42
12	43	42	41	39	40
13	40	39	38	38	38
14	36	37	37	36	35
15	33	33	31	29	30
16	28	27	27	26	28
17	23	22	22	18	22
18	17	16	12	10	11
19	8	7	9	5	2
20	-5	-4	-5	-9	-10

Table 3B: Results of millivolts of ibuprofen in suspension using the potentiometric titration method.

Consumed volume of NaOH (ml)	Millivolt of suspension 1	Millivolt of suspension 2	Millivolt of suspension 3	Millivolt of suspension 4	Millivolt of suspension 5
0	127	123	129	131	136
1	112	112	127	129	126
2	100	98	101	107	105
3	90	90	93	95	96
4	76	74	85	87	89
5	67	68	75	77	79
6	48	50	58	60	65
7	28	30	45	48	53
8	9	18	30	32	28
9	-60	-35	-20	-20	-16

Table 3C: Ibuprofen content in compressed tables using the potentiometric titration method with claimed contents, means, and purity.

Brands	The volume of titrant (ml) based on the potentiometric curve	Measured mg of IBU/tablet	Claimed content (mg)	Relative error	% Purity
Tablet 1	20.50	402.00	400	0.5	100.5
Tablet 2	20.60	404.00	400	1.00	101
Tablet 3	20.60	404.00	400	1.00	101
Tablet 4	20.40	400.00	400	0.00	100
Tablet 5	20.25	397.10	400	-0.725	99.28

Table 3D: Ibuprofen content in suspension using a potentiometric titration method with claimed contents, means, and purity.

Brands	The volume of titrant based on the potentiometric curve	Measured mg of IBU /5 ml	Claimed content (mg)/ 5 ml	Relative error	% Purity
Suspension 1	9.40	92.20	100	-7.8	92.20
Suspension 2	9.50	93.14	100	-6.86	93.14
Suspension 3	9.70	95.10	100	-4.9	95.10
Suspension 4	9.70	95.10	100	-4.9	95.10
Suspension 5	9.70	95.10	100	-4.9	95.10

Table 4A: Results of the pH values of the ibuprofen content in suspension using a pH titration method.

The consumed volume of NaOH (ml)	pH values of Suspension 1	pH values of Suspension 2	pH values of Suspension 3	pH values of Suspension 4	pH values of Suspension 5
0	4.39	4.46	4.42	4.46	4.48
1	5.31	5.38	5.4	5.51	5.71
2	6.11	6.11	6.33	6.58	6.68
3	9.1	9.15	9.14	9.14	9.34
4	10.74	10.73	10.75	10.75	10.65
5	11.16	11.15	11.13	11.13	11.33
6	11.37	11.35	11.38	11.38	11.48
7	11.47	11.47	11.49	11.49	11.52
8	11.58	11.57	11.57	11.57	11.57
9	11.68	11.65	11.65	11.68	11.68
10	11.72	11.72	11.74	11.75	11.75
11	11.77	11.77	11.77	11.8	11.8
12	11.82	11.82	11.83	11.85	11.82
13	11.82	11.85	11.84	11.86	11.83
14	11.83	11.85	11.85	11.87	11.85

Table 4B: Result of pH values of Ibuprofen content in Tablet using a pH titration method.

The consumed volume of NaOH (ml)	pH values of Tablet 1	pH values of Tablet 2	pH values of Tablet 3	pH values of Tablet 4	pH values of Tablet 5
0	4.1	4.12	4.09	4.06	4.04
1	4.43	4.41	4.41	4.41	4.38
2	4.93	4.94	4.95	4.9	4.98
3	5.11	5.12	5.1	5.07	5.05
4	5.59	5.6	5.56	5.58	5.58
5	8.52	8.51	8.51	8.49	8.55
6	10.82	10.79	10.8	10.84	10.82
7	11.47	11.49	11.45	11.48	11.46
8	11.75	11.78	11.74	11.76	11.76
9	11.93	11.9	11.91	11.93	11.93
10	12.2	12.21	12.19	12.17	12.07
11	12.28	12.29	12.27	12.23	12.21
12	12.32	12.35	12.31	12.3	12.3
13	12.45	12.47	12.44	12.42	12.4
14	12.51	12.52	12.5	12.51	12.49
15	12.51	12.55	12.51	12.52	12.5
16	12.54	12.53	12.54	12.51	12.51

Table 4C: Results of d^2pH/dv values of ibuprofen content in suspension using a pH titration method.

The consumed volume of NaOH (ml)	d^2pH/dv values of Suspension 1	d^2pH/dv values of Suspension 2	d^2pH/dv values of Suspension 3	d^2pH/dv values of Suspension 4	d^2pH/dv values of Suspension 5
0	-0.12	-0.19	-0.05	0.02	-0.26
1	2.19	2.31	1.88	1.49	1.69
2	-1.35	-1.46	-1.2	-0.95	-1.35
3	-1.22	-1.16	-1.23	-1.23	-0.63
4	-0.21	-0.22	-0.13	-0.13	-0.53
5	-0.11	-0.08	-0.14	-0.14	-0.11
6	0.01	-0.02	-0.03	-0.03	0.01
7	-0.01	-0.02	0	0.03	0.06
8	-0.06	-0.01	0.01	-0.04	-0.04
9	0.01	-0.02	-0.06	-0.02	-0.02
10	1.77636E-15	1.77636E-15	0.03	-1.77636E-15	-0.03
11	-0.05	-0.02	-0.05	-0.04	-0.01
12	0.01	-0.03	0	0	0.01
13	0.835	0.846428571	0.836428571	0.837857143	0.826428571
14	0.060357143	0.060459184	0.060459184	0.060561224	0.060459184

Table 4D: Results of d^2pH/dv values of ibuprofen content in tablet using a pH titration

The consumed volume of NaOH (ml)	d^2pH/dv values of Tablet 1	d^2pH/dv values of Tablet 2	d^2pH/dv values of Tablet 3	d^2pH/dv values of Tablet 4	d^2pH/dv values of Tablet 5
0	0.17	0.24	0.22	0.14	0.26
1	-0.32	-0.35	-0.39	-0.32	-0.53
2	0.3	0.3	0.31	0.34	0.46
3	2.45	2.43	2.49	2.4	2.44
4	-0.63	-0.63	-0.66	-0.56	-0.7
5	-1.65	-1.58	-1.64	-1.71	-1.63
6	-0.37	-0.41	-0.36	-0.36	-0.34
7	-0.1	-0.17	-0.12	-0.11	-0.13
8	0.09	0.19	0.11	0.07	-0.03
9	-0.19	-0.23	-0.2	-0.18	0
10	-0.04	-0.02	-0.04	0.01	-0.05
11	0.09	0.06	0.09	0.05	0.01
12	-0.07	-0.07	-0.07	-0.03	-0.01
13	-0.06	-0.02	-0.05	-0.08	-0.08
14	0.03	-0.05	0.02	-0.02	0
15	0.75375	#VALUE!	0.75375	-12.5	-12.52
16	0.048984	#VALUE!	0.048984	#VALUE!	12.51

Table 4E: Ibuprofen content in suspension using a pH titration method with claimed contents, means, and purity.

Brands	The volume of titrant (ml) based on the pH curve	Measured mg of ibuprofen / 5 ml	Claimed content (mg) / 5 ml	Relative error	% Purity
Suspension 1	2.55	105.30	100	5.3	105.30
Suspension 2	2.60	107.30	100	7.3	107.30
Suspension 3	2.55	105.30	100	5.3	105.30
Suspension 4	2.55	105.30	100	5.3	105.30
Suspension 5	2.5	103.2	100	3.2	103.2

Table 4F: Ibuprofen content in suspension using a pH titration method with claimed contents, means, and purity.

Brands	The volume of titrant (ml) based on the pH curve	Measured mg of ibuprofen / tablet	Claimed content mg	Relative error	% Purity
Tablet 1	4.8	396.30	400	-0.925	99.10
Tablet 2	4.9	404.54	400	1.135	101.14
Tablet 3	4.8	396.30	400	-0.925	99.10
Tablet 4	4.9	404.54	400	1.135	101.14
Tablet 5	4.9	404.54	400	1.135	101.14

4. Conclusion

In conclusion, all three methods were effective at estimating the ibuprofen content in the analyzed pharmaceutical forms, and the results agreed well with the results of the ibuprofen content in ten brands of compressed tablets and suspensions obtained from various companies via potentiometric, indicator, and second derivative pH titration. All the data were adequate and acceptable to the content claimed on the labels. The pH and potentiometric titration data were compared to the indicator titration data using one-way ANOVA. These methods are easy, economically appropriate, precise, highly accurate, safe, and simple for estimating the ibuprofen content in particular pharmaceutical forms. Additionally, pH and potentiometric titration methods are recommended for determining the ibuprofen content in suspensions, especially when colored suspensions are present. Finally, we recommend that you consider these methods for medical quality control.

5. Conflicts of interest

The author decides that no conflict of interest occurred while conducting this research and writing this article.

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