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Research Article

A STUDY ON ESTABLISHING ANALYTICAL CHEMISTRY PRACTICE ON UHPLC-PDA/MS EQUIPMENT

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ABSTRACT

In this study, an analytical chemistry practice was established and validated via the quantification of ibuprofen in drugs using the UHPLC-PDA/MS analysis for undergraduate students. Accordingly, ibuprofen, extracted from the drug powder, was determined by Acquity UPLC® BEH C18 column (1.7 µm; 2.1 × 50 mm) equipment with a mixture of 0.1% HCOOH:ACN (40:60, v/v) as a mobile phase, and an isocractic flow rate of 0.9 mL min^{-1} at a quantitative wavelength of 254 nm. The data show that the analytical method used possesses high specificity, good linear correlation, and high system suitability. Accordingly, the repeatability (RSD), recovery, limit of detection (LOD), and limit of quantification (LOQ) for this method are 0.75%, 101.3%, 1.00 ppm, and 3.3 ppm, respectively. In particular, the results of the study indicate that the practice is performed at intervals of 4-4.5 hours, with the analytical process reaching the requirements of intermediate precision. In addition, the parameters were further confirmed by the detector of mass spectroscopy (MS) incorporated with the UHPLC device. These findings demonstrate that the analytical procedure is a promising and effective method for establishing the analytical chemistry practice to meet the general education curriculum 2018 in Vietnam and serve as a valuable reference for students and teachers.

Keywords: analytical chemistry practice; ibuprofen; UHPLC-PDA/MS; undergraduate students

1. Introduction

Ultra-high-performance liquid chromatography (UHPLC), a modern instrumental analytical method, is widely utilized. It is an essential tool in basic research and an indispensable tool in environmental analysis and food and drug testing. Hence, practical skills concerning ultra-high-performance liquid chromatography play a vital role in enhancing employment opportunities for students relating to chemistry after graduation. Because of the desire to construct a chemistry practice for undergraduate students, the UHPLC device is employed to reach the requirements, including short analysis time, various

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analytical samples, and standard substances that must be available at the Institute of Drug Quality Control Ho Chi Minh City to facilitate the teaching process. Therefore, this study is conducted to establish an experiment for quantitative analysis of ibuprofen (IBU) drugs in tablets using the UHPLC method.

IBU is a white powder, insoluble in water but soluble in organic solvents with the structural formula shown in Figure 1 (British Pharmacopoeia Commission, 2022a; United States Pharmacopeial Convention, 2020a; Vietnam Ministry of Health, 2017a). IBU is a non-steroidal anti-inflammatory drug with analgesic, antipyretic, and anti-inflammatory effects. In Vietnam, this drug is widely circulated in many forms of preparations, such as topical creams, intravenous drugs, capsules, and especially tablets (Vietnam Ministry of Health, 2018).

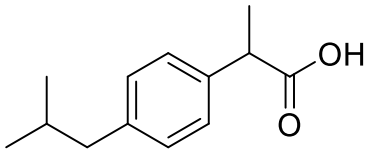

Figure 1. *Structure of ibuprofen*

Therefore, it is convenient to choose the IBU tablets as analytical samples. The IBU content in medicinal preparations and plasma was analyzed by many methods such as HPLC (Ali et al., 2012; Aravind et al., 1984; Chen et al., 2016; Cunha et al., 2015; Koçak & AtiLa, 2022; Nakov et al., 2016), high-performance thin-layer chromatography (HPTLC) (Padró et al., 2016; M. Ragab et al., 2014; M. A. A. Ragab et al., 2019; Shah et al., 2014), capillary electrophoresis (Cunha et al., 2015) and spectrofluorimetry (Damiani et al., 2001). Nowadays, the Vietnam Pharmacopoeia (VP), the United States Pharmacopoeia (USP), and the British Pharmacopoeia (BP) have their monographs for the quantification of IBU in the tablets by HPLC (British Pharmacopoeia Commission, 2022b; United States Pharmacopeial Convention, 2020b; Vietnam Ministry of Health, 2017b). In general, these procedures require a prolonged interval for sample preparation and chromatographic analysis. To overcome this obstacle, the Thermo Scientific company has collaborated with Grosse et al. to conduct research to improve the analytical process of IBU in tablets based on the USP 40 standard. With the use of the UHPLC-PDA/MS method, the study reduced the chromatographic analysis time to 16 times (from 8 to 0.5 min) and saved more than 97% of mobile phase solvent volume (Grosse et al., 2018). However, the sample preparation procedure has not been significantly enhanced. Therefore, in our research, controlling the chromatographic conditions combined with available facilities can improve the sample preparation process, validate the analysis procedure, and conduct an experiment based on the developed procedure. This is a new approach for designing the analytical chemistry practice relating to the knowledge of chromatography and mass spectroscopy to meet the requirements for the new general education program 2018 in Vietnam. Then the results of this study can be a useful source for students and teachers.

2. The experiment

2.1. Chemicals and instruments

The IBU standard (99.5%, Lot No. QT026 130520) was purchased from the Institute of Drug Quality Control in Ho Chi Minh City (Vietnam). Acetonitrile (99.95%) for UHPLC was supplied by Prolabo (USA), and formic acid (98%, reagent grade) was provided by Scharlau (Spain). The drug sample used in this study is 400 mg IBU tablets (Nadyphar, Lot No. 21004B) circulating in the market and purchased at pharmacies in Ho Chi Minh City.

Chromatographic analysis was performed on an Acquity UPLC® H-Class Plus chromatograph system supplied by Waters. This system was operated by the Acquity UPLC® BEH C18 column (1.7 µm, 2.1 × 50 mm, P/N 186002350), Acquity UPLC® eλ PDA detector, Waters SQ Detector for MS, and controlled by Masslynx V4.2 software. In addition, this study used analytical balance (Shimadzu AUW220, ± 0.1 mg), centrifuge machine (Hettich Zentrifugen D-78532 Tuttlingen), and ultrasonic bath (Elmasonic S 100 H).

2.2. Research methods

2.2.1. Investigation of chromatographic conditions

To accommodate experimental conditions, we changed some chromatographic conditions from the procedure developed by Grosse *et al.* (2018). Accordingly, we used a chromatographic column with the same size but reduced the particle diameter from 2.6 to 1.7 µm. At the same time, the mobile phase was utilized as H_2O:ACN (40:60, v/v), 0.4% chloroacetic acid with 0.1% HCOOH:ACN (40:60, v/v). Because of the above adjustment, we reinvestigated the chromatographic conditions with the initial condition, including the Acquity UPLC® BEH C18 column (1.7 µm; 2.1 × 50 mm), 10 µL of the injection volume, the mixture of 0.1% HCOOH:ACN (40:60, v/v) as a mobile phase, an isocratic flow rate of 0.6 mL min^{-1} and a quantitative wavelength of the PDA detector at 254 nm. Besides, the MS detector is set to ESI^- mode with a capillary voltage of 2.9 kV, a cone voltage of 22 V, an RF lens voltage of 2.5 V, and an extractor voltage of 2 V at a source temperature of 130°C. Further, the desolvation temperature, cone gas flow, and desolvation gas flow are 400°C, 60 $L{\cdot}h^{-1}$, and 600 $L{\cdot}h^{-1}$, respectively.

2.2.2. Preparation of the standard solution

The IBU stock standard solution (10 $mg{\cdot}mL^{-1}$) was prepared by introducing 100.5 mg of the IBU standard into 10 mL of ACN. This stock standard solution is stored at 4°C and used to dilute the lower concentrations of the IBU solutions. The lower concentration of the IBU solutions, including 7.5, 5.0, 2.5, and 1.0 $mg{\cdot}mL^{-1}$ IBU solutions prepared by dilute 3.75, 2.50, 1.25, and 0.50 mL of stock standard solution in 5.00 mL volumetric flask with ACN, respectively. These solutions were filtered through a 0.22 µm membrane filter before injection into the chromatographic system.

2.2.3. Preparation of the sample

A sample preparation procedure to optimize the analysis time is suggested as follows: Twenty tablets containing IBU were ground into the powder. Then, 40 mg of tablet powder containing IBU was ultrasonicated in 5 mL of ACN solvent for 5 minutes. The mixture was centrifuged to remove the insoluble components, in which the solution was transferred to a volumetric flask (10 mL). Subsequently, the redundant solid was rinsed about two times with ACN (2 mL per time) to ensure complete extraction of IBU from tablets before being added to the above volumetric flask. Finally, the solution was filtered through a 0.22 μm membrane filter and injected into the chromatographic system, respectively. The following equation calculated the IBU content:

$$\text{IBU content (\%)} = \frac{A-b}{a} \times V \times \frac{\text{drug content}}{\text{tablet content}} \times \frac{100\%}{\text{labeled amount}} \quad (1)$$

Where A (μAU·min), a, and b are the peak area, the slope, and the intercept of the standard curve, respectively. V (mL) is the solution volume contained in the volumetric flask. Drug content (mg) is the mass of the used drug powder. Tablet content (mg) is the average mass of a tablet, and labeled amount (mg) is the amount of IBU in a tablet as indicated on the label.

2.2.4. Validation

After the optimized condition, the analytical procedure was validated in line with the guidance of Appendix 8 in the Drug Registration Handbook issued by the Vietnam Ministry of Health, USP 43 and Vietnamese Pharmacopeia V (United States Pharmacopeial Convention, 2020b; Vietnam Ministry of Health, 2013, 2017b). The validation parameters include suitability, specificity, linearity, limit of detection, limit of quantification, precision, and accuracy of the system.

2.2.5. Experiments

The experiments were conducted on four groups of fourth-year students majoring in chemistry and chemistry education at Ho Chi Minh City University of Education in Vietnam. The obtained results were used to evaluate the feasibility of the practice in terms of intermediate precision and organization time and to identify any issues during teaching.

3. Results and discussion

3.1. Investigation of chromatographic conditions

3.1.1. Investigation of the flow rate

The IBU solution with 10 mg·mL^{-1} concentration was analyzed on UHPLC equipment using the PDA detector at a wavelength of 254 nm with various flow rates from 0.6 to 1.1 mL·min^{-1}. The collected data revealed that the system pressure exceeded the instrument's limit at the flow rates over 1.0 mL·min^{-1}. At the flow rates of less than 1.0 mL·min^{-1}, the

chromatogram indicated that the IBU peak was completely separated from the solvent peak, but a double peak was observed, as shown in Figure 2.

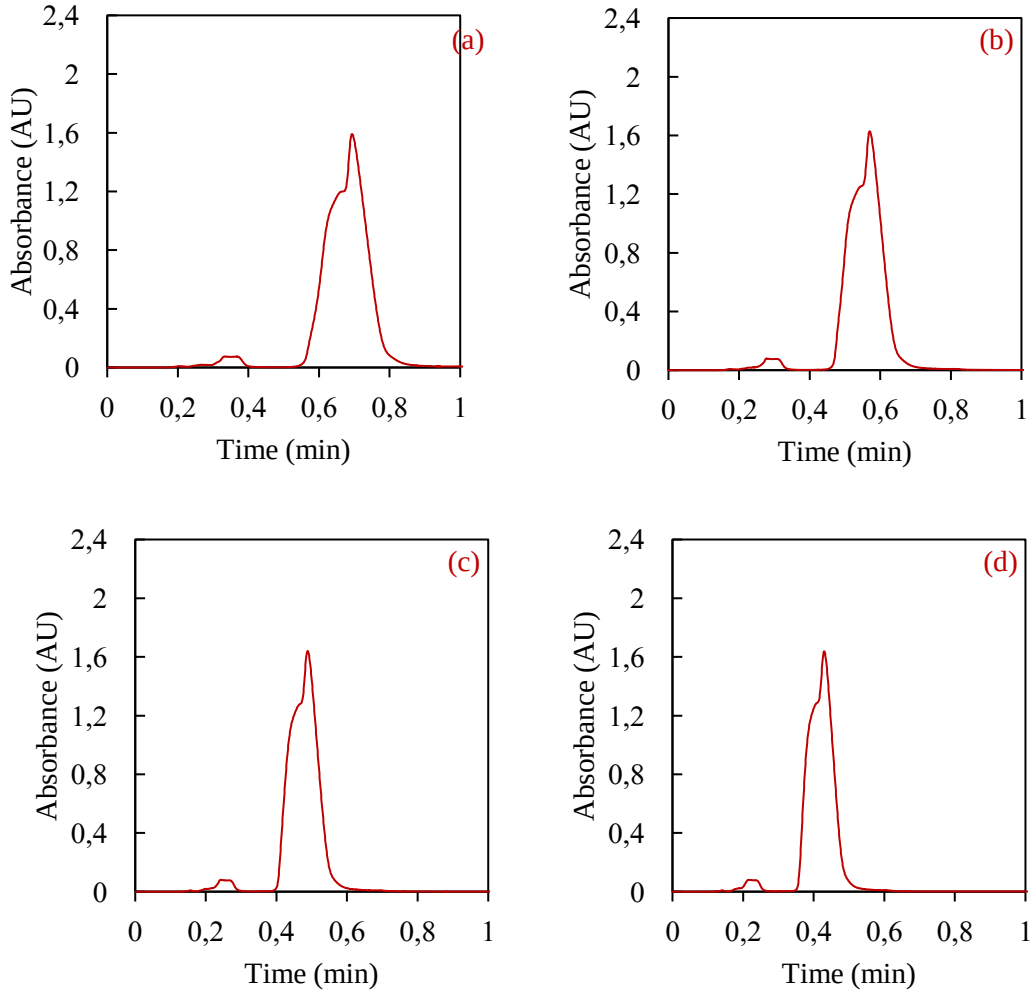

Figure 2. *The chromatograms of IBU at different flow rates: (a) 0.6 mL·min^{-1}; (b) 0.7 mL·min^{-1}; (c) 0.8 mL·min^{-1}; and (d) 0.9 mL·min^{-1}*

The UV spectra at the retention times of 0.40 and 0.43 min in the chromatograms at 0.9 mL·min^{-1} flow rate are exhibited in Figure 3. The uniformity of the shape in the UV spectra shows that the appearance of a minor chromatographic peak in front and partial overlap of the prominent chromatographic peak belongs to the same IBU analyte, which is not due to either impurities or degradation products of IBU. To cope with this unexpected peak splitting, we will investigate the effect of the sample injection volume at a selected flow rate of 0.9 mL·min^{-1} in the next section.

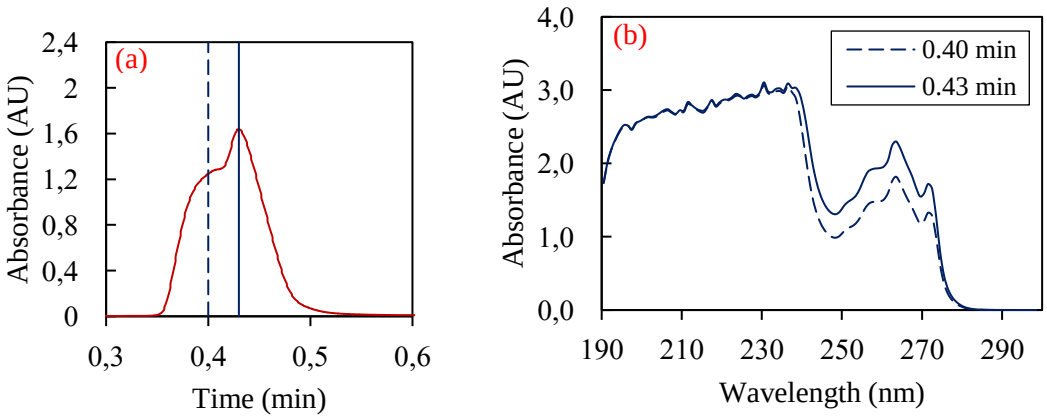

Figure 3. *The chromatograms of IBU at the flow rate of 0.9 mL·min^{-1} (a) and the UV spectra of IBU at the different retention times of 0.40 and 0.43 min (b)*

3.1.2. Investigation of the injection volume

The IBU solution (10 mg·mL^{-1}) at the different injection volumes from 0.5 to 10 µL is performed at an optimal flow rate of 0.9 mL·min^{-1}. The chromatograms are recorded and clearly shown in Figure 4. The results show that the double peaks are found as the injection volume exceeds 5 µL. This can be explained by the fact that IBU dissolved in pure ACN differs from the mobile phase, a mixture of ACN with 0.1% HCOOH solution in water. After injecting the sample into the system, the analyte is mixed with the dynamic phase, forming a region with the excess ACN solvent. With a general reverse phase liquid chromatography method, it is noted that the dynamic phase region with the excess less polar ACN possesses a stronger eluent strength, resulting in partial elute of the analyte from the column before complete elute. This causes the appearance of a smaller minor chromatographic peak immediately preceding and partially covering the main chromatographic peak in the chromatogram. Furthermore, the slow equilibration of the analyte between the two phases can lead to the reported problems. Consequently, the mass transfer rate of the analytes between the two phases is slower than that of the mobile phase at high flow rates and injection volumes, leading to the broadening and splitting of the chromatographic signals. As illustrated in Figure 4, it is interesting to note that the chromatographic peak is split when the injection volume is large. Meanwhile, the injection volume is too small, leading to the tailing of the chromatographic peak. However, the shape of the chromatographic peak obtained at an injection volume of less than or equal to 5 µL is acceptable. Hence, the injection volume of 1 µL is selected for the following studies to gain the perfect chromatographic shapes and limit the effect of the high amount of analyte.

In this study, the chromatographic conditions, such as the Acquity UPLC® BEH C18 column (1.7 µm, 2.1 × 50 mm), the injection volume of 1 µL, the mobile phase of 0.1% HCOOH:ACN (40:60, v/v) mixture, the isocratic flow rate of 0.9 mL·min^{-1}, and quantitative wavelength of PDA detector at 254 nm are optimal to quantitate the IBU content in the tablets effectively.

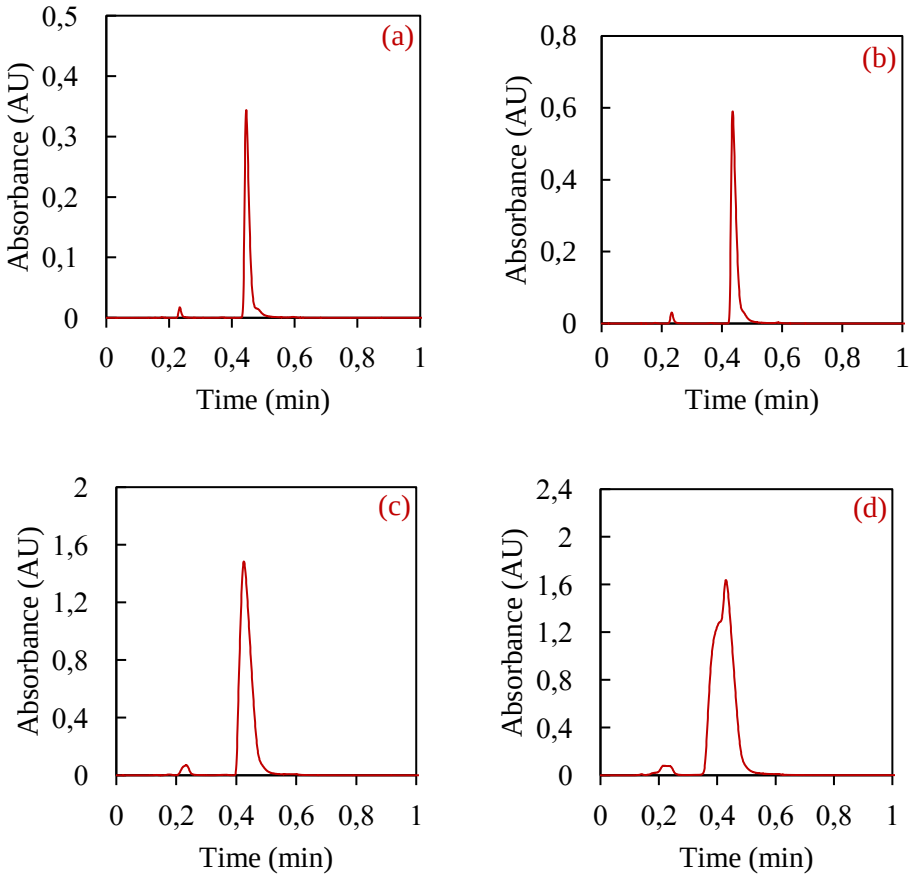

Figure 4. *The IBU chromatograms at different injection volumes: (a) 0.5 µL; (b) 1.0 µL; (c) 5.0 µL; and (d) 10 µL*

3.1.3. The extraction efficiency

Based on the proposed procedure, the IBU extractions were fabricated by dissolving various dosages of the IBU powder in ACN solvent. Each extraction was injected into the chromatographic system. The results show that about 99.7% of IBU can be extracted with just one extraction (Table 1). Thus, the proposed extraction procedure of IBU in powder is efficient with a high separate level.

Table 1. *The results of the extraction efficiency*

Drug content (mg)	**Times**	**Retention time (min)**	**Peak area (µAU·min)**
67.1	1	0.443	4149.702
	2	0.448	9.954
	3	0.448	3.110
135.7	1	0.439	8216.503
	2	0.448	19.991
	3	0.448	4.051

3.2. Validation

3.2.1. The suitability of system

The IBU standard solution was injected into the chromatographic system with six injection times under the selected conditions. As displayed in Table 2, the values of retention time, peak area, tailing factor, and resolution of two peaks at 0.44 min and 0.23 min (RSD) are < 2%. In addition, the mean tailing factor is less than 2.5. Thus, the analytical method meets the general requirements for system suitability.

Table 2. *The results of the system suitability*

No.	Retention time (min)	Peak area (µAU·min)	Tailing factor	Resolution
1	0.441	10704.919	1.94	2,02
2	0.442	10602.175	1.88	2,04
3	0.440	10633.048	1.91	1,99
4	0.441	10562.722	1.90	2,02
5	0.441	10602.660	1.86	1,99
6	0.441	10601.903	1.89	1,97
Mean =	0.441	10617.905	1.90	2,00
SD =	0.001	48.13	0.03	0,03
RSD =	0.12%	0.45%	1.4%	1,3%

3.2.2. Specificity

The blank of ACN solvent, the IBU standard solution, the sample, and the spiked sample were injected into the chromatographic system. Herein, the IBU standard solution concentration is chosen to be 5 $mg \cdot mL^{-1}$ to efficiently compare the specificity of the method with the analytical sample (about 4 $mg \cdot mL^{-1}$). It is noted that no response peak coincided with the retention time of the standard solution. In contrast, the standard solution, the sample, and the spiked sample possess high response peaks at similar retention times. At the same time, the peak of the spiked sample has a larger area than the sample because the spiked sample has a more significant concentration of IBU. Therefore, the analytical method in this study has a high specificity. The chromatograms of blank and sample are shown in Figure 5.

Table 3. *The results of the specificity*

No.	Injection sample	Retention time (min)	Peak area (µAU·min)
1	Blank	Not responding	-
2	Standard solution	0.443	5682.418
3	Sample	0.444	4105.266
4	Spike sample	0.445	5216.077

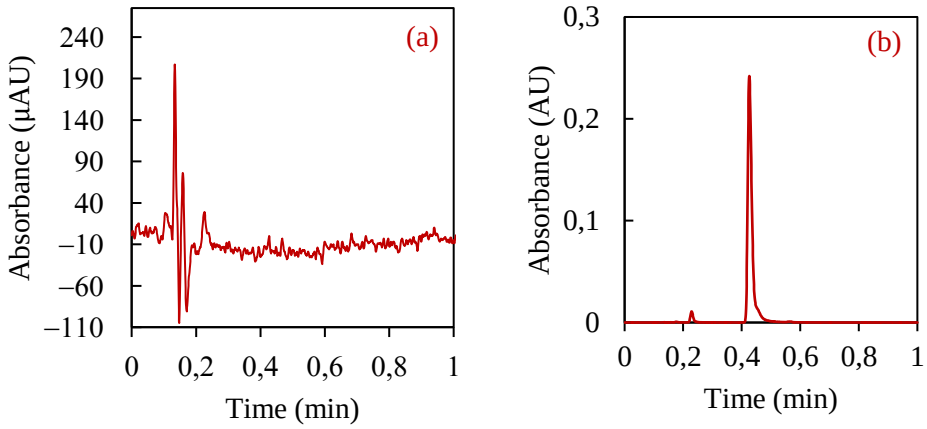

Figure 5. *The chromatograms of the blank (a) and sample (b)*

3.2.3. Linearity

The standard solutions were prepared with the IBU concentrations from 1 to 10 mg·mL^{-1} and injected into the chromatographic system. The data are shown in Table 4 and Figure 6. The regression results for the coefficient value of $R^2 = 0.9994 > 0.9980$. Thus, there is an excellent linear dependence between the chromatographic peak area, and the concentration of IBU ranges from 1 to 10 mg·mL^{-1}.

Table 4. *The results of the linearity*

No.	IBU concentration (mg·L^{-1})	Mean peak area (µAU·min)	Regression
1	1000	1175.346	
2	2500	2791.317	$y = (1.028 \pm 0.014)x + (221 \pm 86)$
3	5000	5515.503	
4	7500	7900.816	$(R^2 = 0.9994)$
5	10000	10461.826	

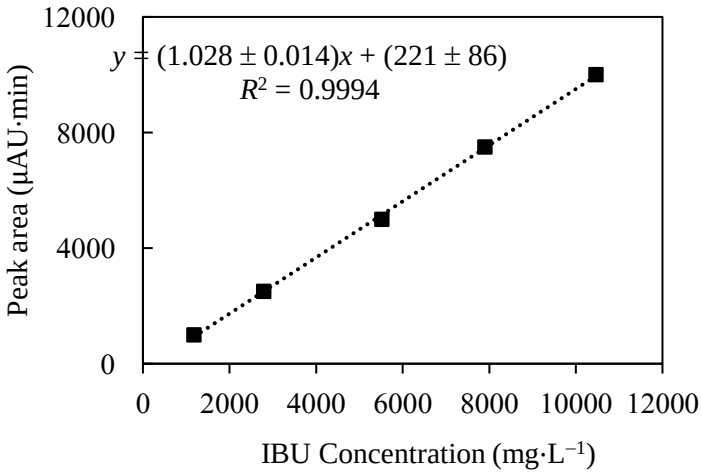

Figure 6. *The IBU standard curve with different concentrations from 1 to 10 mg·mL^{-1}*

3.2.4. Limit of detection and limit of quantitation

The standard IBU solution was diluted and injected into the chromatographic system. The gained data confirm that the S/N ratio is approximately 3 at a low concentration of 1.00 $mg \cdot L^{-1}$, and the limit of detection (LOD) reaches a value of 1.00 $mg \cdot L^{-1}$. Hence, the limit of quantification is determined according to the following formula:

$$LOQ = \frac{10}{3} \times LOD = \frac{10}{3} \times 1.00\ mg \cdot L^{-1} = 3.33\ mg \cdot L^{-1}$$

3.2.5. Repeatability

We analyzed six independent samples of the same drug types based on the observed method. The results are exhibited in Table 5. The method's relative standard deviation (RSD) is less than 2.0%. Therefore, the method meets the requirements of repeatability. In addition, the obtained parameters reveal that the content of IBU in the drug is (96.0 ± 0.8)% within the acceptance criterion of Vietnam Pharmacopoeia V, which is from 95.0% to 105.0% (Vietnam Ministry of Health, 2017b).

Table 5. *The results of the repeatability*

No.	Drug dosage (mg)	Mean peak area (µAU·phút)	IBU content (%)	Data processing
1	67.5	4182.773	96.2	Mean = 96.0
2	67.5	4201.624	96.6	SD = 0.72
3	67.4	4177.663	96.2	RSD = 0.75%
4	67.5	4202.152	96.7	Confidence interval = 96.0 ± 0.8
5	67.1	4119.701	95.2	(P = 95%)
6	67.1	4109.700	95.0	
Tablet content = 674.2 mg				

3.2.6. Trueness

Table 6. *The results of the trueness*

No.	Drug content (mg)	Amount of IBU (mg)			Recovery (%)
		Available	Spike	Found	
1	67.7	38.9	10	49.1	102.7%
2	67.7	38.9	10	49.0	101.2%
3	67.7	38.9	10	48.9	99.9%
				Mean =	101.3%
				SD =	1.11
				RSD =	1.1%

Table 6 displays the trueness of the procedure investigated on the three samples and the three spiked samples. The results show that the RSD and recovery values are < 2.0% and 101.3%, respectively, which are suitable with the general analytical criterion (from 98.0% to 100%) (Vietnam Ministry of Health, 2013).

3.2.7. Validation by the UHPLC/MS method

Figure 7a indicates the mass spectrum of IBU with the EIS⁻ mode. It is noted that the primary peak of IBU is at m/z = 205.08, corresponding to the $[M–H]^-$ ion. Accordingly, the UHPLC/MS method was conducted with the 500 ppb IBU solution diluted from the IBU stock standard solution at a flow rate of 0.9 mL·min^{-1}, the injection volume from 1 to 10 µL, with an aforementioned mobile phase system. As shown in Figure 7b, the chromatographic peak intensity is proportional to the injection volume. In particular, when the injection volume is increased, the chromatographic peak is not split, which is different from the results from the previous UHPLC-PDA analysis in this work.

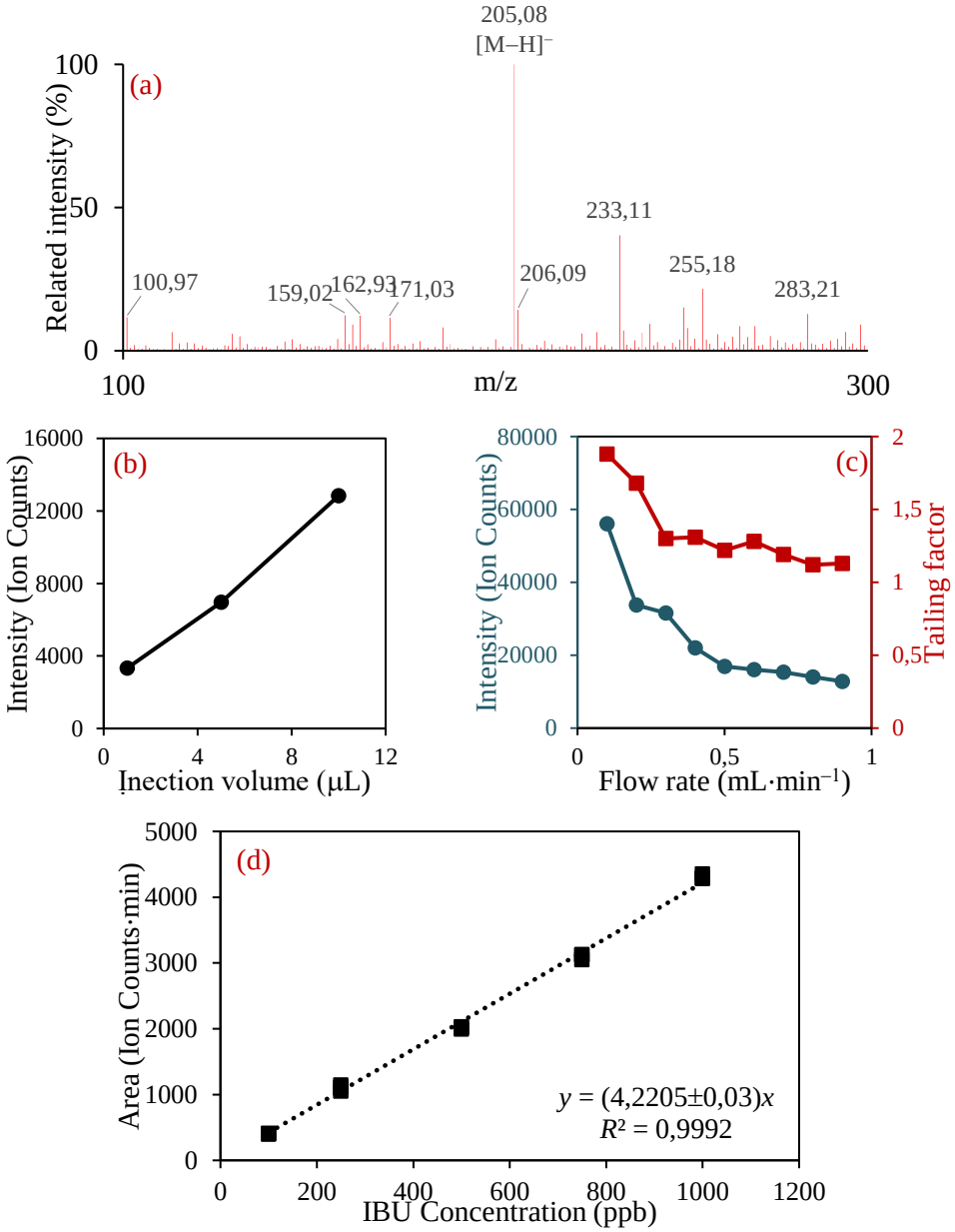

Figure 7. *The results obtained from the UHPLC-MS analysis*

To further gain insight into the effect of the flow rate on the peak intensity and the tailing factor, the IBU solution of 500 ppb was injected into the chromatographic system with the injection volume of 10 µL at the various flow rates from 0.1 to 0.9 mL·min^{-1} and the chromatogram was recorded based on the signal intensity of the $[M–H]^-$ ion. The results in Figure 7c reveal that both the chromatographic peak's signal intensity and tailing factor increase as the flow rate decreases. This can be explained by the fact that when the flow rate decreases, the infusion rate of the MS probe also decreases correspondingly, driving the increase of ionization efficiency and signal intensity. However, when the flow rate decreases, the IBU molecules will be located at the chromatographic column with a longer interval, which broadens the chromatographic peak and increases the tailing factor. Moreover, as the flow rates decreased from 0.9 to 0.3 mL·min^{-1}, there was no significant change in the tailing factor but a remarkable increase as the flow rate dropped below 0.3 mL·min^{-1}. Therefore, the MS detector's optimal flow rate is 0.3 mL·min^{-1}. Additionally, at a flow rate of 0.3 mL·min^{-1} and injection volume of 10 µL, the MS detector indicates an excellent linear dependence between the chromatographic peak range and the IBU concentration at the range of 100-1000 ppb, which is much lower than that of the PDA detector as illustrated in Figure 7d.

3.3. The experiments

3.3.1. Intermediate precision

From the above-developed analytical method, we designed a chemistry practice and conducted experiments on four groups of fourth-year students at Ho Chi Minh City University of Education. The experimental data are summarized in Table 7 and Table 8.

Table 7. *The results of the experiments*

Group	Standard curve equation	IBU contents (%)				RSD
		1st	2nd	3rd	Mean	
1	$y = 1098.2 \times x - 17.6$ ($R^2 = 0.9988$)	93.0	94.9	96.9	94.9	2.0%
2	$y = 1107.7 \times x - 59.5$ ($R^2 = 0.9967$)	94.2	96.4	96.9	95.9	1.5%
3	$y = 1155.4 \times x + 92.2$ ($R^2 = 0.9990$)	94.7	96.7	93.8	95.1	1.6%
4	$y = 1096.1 \times x + 150.3$ ($R^2 = 0.9986$)	94.7	95.3	96.9	95.6	1.2%

Table 7 shows that the results of each group have RSD < 2.0% (except for group 1). This reveals that the data have good internal repeatability. Noteworthily, the relative standard deviation for group 1 is approximately acceptable. This can be accounted for the emerged errors during the sample preparation, such as spillage of the extract causing loss of samples and reduced repeatability between the analyses.

Table 8. *ANOVA tests*

Source of variation	*df*	*SS*	*MS*	
Between groups:	$f_1 = 4$	SSG = 3.18	MSG = 0.80	$F = 0.49$
Within groups:	$f_2 = 13$	SSE = 21.2	MSE = 1.63	$F_{crit} = 3.18$

To evaluate the intermediate precision, we compared the results of four groups with our previous data with ANOVA tests. Table 8 shows that the value of F is lower than F_{crit}, indicating no significant difference in the results between our method and the experiments and among the experiment groups. Following these achievements, the analytical method serves the general requirements with a reliable intermediate precision level.

3.3.2. The common problems

During the pedagogical experiment, we noticed some the common problems as below:

(i) Firstly, regarding the organization time, the time to conduct the lesson with the lecturer and students' practice (two groups) was about 4.5-5 hours. This time is equivalent to a class session. However, it is necessary to rearrange the learning activities so that students have more time to study and operate on the UHPLC device.

(ii) Secondly, regarding practical skills, most students needed help using micropipettes, especially in experiments with organic solvents such as the ACN solvent. Specifically, in transferring the extract from the centrifuge tube to the volumetric flask with a Pasteur pipette, the students often spilled the extract. This phenomenon occurred because the ACN solvent has a slight surface tension, leading to a weak hold of the liquid droplets at the tip of the pipette, making the liquid drops at the tip of the pipette easy to fall.

(iii) Thirdly, regarding the operation of the chromatographic system, most students often needed help with the stage of chromatographic sample declaration, such as naming the same name and declaring the wrong vial position. In particular, most students often ran the sample right after placing the sample in the automatic sampler without waiting for the temperature stabilization time, leading to significant errors in the repeatability of the peak area compared to the initial analysis.

4. Conclusion

In conclusion, this work developed and validated a quantification procedure of the IBU content in the tablets using the UHPLC-PDA instrument combined with the MS detector. Following the analytical method conducted, we established and designed an analytical chemistry practice for undergraduate students relating to chemistry. The validation results show that the proposed analytical method has high specificity, good linear correlation, systematic compatibility, repeatability, and trueness and meets the acceptable requirements. Furthermore, the pedagogical experiment data reveal that the procedure meets the reasonable requirements of intermediate precision and appropriate class organization time. These results show that this study opens up a new investigation direction concerning chromatography and mass spectroscopy to respond to the general education program 2018 in Vietnam and as a helpful source for researchers.

❖ ***Conflict of Interest:*** *Authors have no conflict of interest to declare.*

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XÂY DỰNG BÀI THỰC HÀNH HOÁ HỌC PHÂN TÍCH TRÊN THIẾT BỊ UHPLC-PDA/MS

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TÓM TẮT

Nghiên cứu này xây dựng và thẩm định một bài thực hành hoá học phân tích về định lượng ibuprofen trong viên nén bằng phương pháp UHPLC-PDA/MS cho các sinh viên ở bậc đại học. Theo đó, ibuprofen được trích li từ bột thuốc và thực hiện phân tích sắc kí bằng cột Acquity UPLC® BEH C18 (1.7 µm; 2.1 × 50 mm) với hệ phase động là hỗn hợp 0.1% HCOOH:ACN (40:60, v/v), tốc độ dòng đẳng môi là 0.9 mL/min và định lượng ở bước sóng 254 nm. Kết quả thẩm định cho thấy phương pháp phân tích có tính đặc hiệu cao, có tương quan tuyến tính và tương thích hệ thống tốt, độ lặp lại RSD = 0.75%, độ thu hồi đạt 101,3%, giới hạn phát hiện LOD là 1.00 ppm và giới hạn định lượng LOQ là 3.30 ppm. Kết quả thực nghiệm sư phạm cho thấy thời gian thực hiện bài thực hành khoảng 4.5-5 giờ và quy trình phân tích đạt yêu cầu về độ chụm trung gian. Ngoài ra, các thông số thực nghiệm trên còn được xác nhận bằng đầu dò khối phổ (MS) kết hợp với thiết bị UHPLC. Những tìm thấy này đã chứng minh rằng phương pháp phân tích được sử dụng là một phương pháp đầy hứa hẹn và hiệu quả cho việc xây dựng bài thực hành hóa học phân tích đáp ứng chương trình giáo dục phổ thông 2018 và là nguồn tài liệu tham khảo có giá trị cho sinh viên và giáo viên.

Từ khóa: bài thực hành hoá học phân tích; ibuprofen; UHPLC-PDA/MS; sinh viên đại học