Food Research 4 (5) : 1758 - 1766 (October 2020)
Journal homepage: http://www.myfoodresearch.com

FOOD RESEARCH

FULL PAPER

Determination of acid, peroxide, and saponification value in patin fish oil by FTIR spectroscopy combined with chemometrics

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Article history:
Received: 16 January 2020
Received in revised form: 17 March 2020
Accepted: 22 March 2020
Available Online: 15 April 2020

Keywords:
Acid value,
Peroxide value,
Saponification value,
Patin fish oil,
FTIR,
Chemometrics

DOI:
https://doi.org/10.26656/fr.2017.4(5).030

Abstract

Simple, rapid, and reproducible methods for determining the acid value (AV), peroxide value (PV), and saponification value (SV) of patin fish oil (PFO) were developed using Fourier Transform Infrared (FTIR) spectroscopy combined with chemometrics of Principal Component Regression (PCR) and Partial Least Square (PLS). The relationship between actual values was determined using AOCS method and predicted value was determined with FTIR spectroscopy and chemometrics. From the validation work, the high coefficient of determination (R^2) reached up to > 0.99. This study concluded that by means of FTIR spectra that combined with PCR and PLS technique can be used to determine AV, PV, and SV of PFO.

1. Introduction

Patin (*Pangasius micronemus*) is a freshwater fish in Indonesia and have exported for years to the western market. Indonesia is the second largest of patin producer, with production reaching up to 16.1% of the total production of patin in the world (Ramadhan *et al*., 2016). Hashim *et al.* (2015) have sucessfully extracted fatty acid from patin and get lipid up to 31% with the level of omega-3 was 4.7%. Hence, patin can be a good source of omega-3. While in Thailand and Vietnam, patin was extracted to obtain the fish oils and the patin fish oils (PFO) have been widely traded.

In the market, that is important to know the quality of fish oil. Some parameters used to determine the quality of fish oil. To determine the quality of fish oil can be performed by determining of physico-chemical values such as acid, peroxide, and saponification value (Rohman *et al*., 2015). Titration method was used to determine the acid, peroxide, and saponification value, based on the chemical reaction between free fatty acids and the reagent. But, the titration method has some disadvantages such as being time-consuming and labor-intensive, requiring large amounts of organic solvents, use of the highly toxic, and carcinogenic reagent, difficulty in distinguishing the end-point with samples containing coloured substances and largely dependent on the skills of the analyst (Rohman *et al*., 2012; Jiang *et al*., 2016).

Currently, Fourier transform infrared spectroscopy (FTIR) combined with chemometrics has been widely used for determining acid, peroxide, and saponification value. FTIR has been used for determination of acid value on edible oil-based on -O-H stretching (Jiang *et al*., 2016), peroxide value on edible oils based on -COO- stretching (Yu *et al*., 2009; Hu *et al*., 2019), to monitoring peroxide value in oxidized emulsions (Hayati *et al*., 2005), and saponification value on red fruit oil using partial least square calibration (Rohman *et al*., 2015).

However, the application of FTIR spectroscopy for the determination of acid, peroxide, and saponification value of patin fish oil (PFO) has not been reported yet. Therefore, FTIR spectroscopy combined with principal component regression (PCR) and partial least square (PLS) was developed for the determination of acid, peroxide, and saponification value of PFO from some

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eISSN: 2550-2166

extraction methods.

2. Materials and methods

2.1 Materials

Patin from Tulungagung, East Java, Indonesia. N-hexane, chloroform, ethyl acetate, ethanol, HCl, acetic acid, sodium thiosulphate, KI, acetone, KOH were obtained from Merck (Germany), phenolphthalein indicator and starch from Progo Mulyo store in Yogyakarta.

2.2 Extraction method

Extractions of PFO were performed using maceration method with wet and dried samples and soxhlet method also with three variations of solvent are n -hexane, ethyl acetate, and chloroform. The patin flesh was cutting into small pieces then extracted with the solvent for maceration with wet samples. Patin flesh was dried in the oven at 60°C for 24 hrs before extracted using maceration with dried samples and Soxhlet method. Maceration process was performed at room temperature while the Soxhlet method at 82°C. After the extraction step, the solvent was evaporated using the rotary evaporator at 50°C to get the PFO.

2.3 Acid value determination

Determination of acid value using the AOCS official method Cd 3a–63 (AOCS, 2004). The oil sample (2 g) was mixed with 20 mL ethanol and added with 2 mL phenolphthalein in a 250 mL Erlenmeyer flask. The mixture was titrated with 0.01 M of KOH and shaken vigorously until the color was changed (from white to pink). The result was expressed as the number of milligrams of KOH required to neutralize the free fatty acid in 1 g of the sample.

2.4 Peroxide value determination

Peroxide value is a measure of peroxides contained in the oil and is determined by measuring iodine released from potassium iodide. Determination of peroxide value was performed according to AOCS official method Cd 8b-90 (AOCS, 2005). The oil sample (5±0.01 g) was dissolved in 30 mL acetic acid-chloroform (3:2) solution. Then saturated KI solution and distilled water were added and shake the flask vigorously to liberate iodine from chloroform layer. The mixture was titrated with 0.01 N sodium thiosulphate using starch solution as an indicator.

2.5 Saponification value determination

The saponification value was expressed as the number of milligrams of potassium hydroxide (KOH) required to saponify 1 g of oil. Determination of saponification value was according to AOCS method Cd 3-25 (AOCS, 1990). The 2 g of oil were dissolved with ethanol in Erlenmeyer flask. Then connected with an air condenser and boil gently for 1 hr in order that sample is completely saponified. After it cooled and added 1 mL phenolphthalein. The mixture was titrated with 0.5 N HCl until the pink colour has just disappeared.

2.6 FTIR analysis

Samples were analysed using FTIR spectrometer (Thermo Scientific Nicolet iS10, Madison, WI) using Omnic software. The measurements were done in the middle infrared region of 650-4000 cm^{-1} with 32 scans and at the resolution was 16 cm^{-1}. The background scan was performed to reduce the effect of the reference spectrum of the air. Before and after analysis sample, ATR crystal was cleaned with acetone p.a. Replications were done with scanning the same samples for three times.

2.7 Data analysis

Multivariate analysis of the acquired data was performed using principal component regression (PCR) and partial least square (PLS) with TQ Analyst software version 9 (Thermo Fisher Scientific Inc.) while the statistic analysis was done using Minitab 18. In this research, Principal component regression (PCR) and partial least square (PLS) were used to determine the physico-chemical value from FTIR spectral. PCR is one of inverse calibration technique in which the physico-chemical value from titration method (x-axis) is used as a predictor, while responses such as absorbance at several wavelengths are located on the y-axis. PCR performs multiple inverse calibrations of predictor variables against the scores (knowns as principal components) rather than original variables (Rohman and Putri, 2019). Whereas the PLS method uses a correlation between changes in spectral absorption and sample concentration then computing with other spectra that can disturb the analyte spectra (Ballabio and Todeschini, 2009). In this research using normal, first derivative and second derivative spectra to identify the targeted spectra from the compounds by increasing the spectral resolution (Putri *et al*., 2019).

The collected data set was divided into calibration and validation subset. Prediction models were validated with cross validation 'leave one out' technique. Cross validation evaluates the data by excluding selected samples in the regression model and then constructing a new model for the remaining samples. The new model was evaluated and the error values for predicted observations are computed. The new samples are then

excluded from the model set and a new model is constructed. This procedure is repeated until all samples in the PCR and PLS models have been excluded once (Rohman and Che Man, 2010). In order to compare the performances of the developed calibration models and express their predictive ability, two statistical values, namely root mean square error of calibration (RMSEC) and root mean square error of prediction (RMSEP) were calculated. Among these values, RMSEP gives the best estimation of future performance of the calibration model (Temiz *et al*., 2017).

3. Results and discussion

3.1 FTIR Spectra of PFO

The FTIR spectra of PFO was shown in Figure 1. Each peak corresponded to a functional group of fatty acid structure. The peak at wavenumber 721 cm^{-1} was due to the rocking vibration of methylene ($-CH_2$), while the peaks at 1114 and 1234 cm^{-1} were from -C-O vibrations. The bending vibration of methylene was observed at wavenumber 1461 cm^{-1}. Meanwhile the carbonyl (C=O) stretching vibration was observed at a wavenumber 1744 cm^{-1}. The peaks at wavenumbers 2854 and 2921 cm^{-1} represent asymmetric and symmetric stretching vibration of methylene ($-CH_2$) respectively (Rohman *et al*., 2011; Putri *et al*., 2019).

3.2 Determination of AV

The acid value (AV) is an explanatory parameter for evaluation of the level of hydrolysis of oil. Free fatty acid and glycerol were the results of the hydrolysis reaction. The acid value usually can be determined by acid/base titration (Mahboubifar *et al*., 2016). The AV from both titration and FTIR methods were shown in Table 1. The AVs from FTIR were obtained from optimization method (Table 2). Based on the ANOVA test using Minitab 18, p-value from both methods was 0.00. So, the result from both methods not significantly different ($p<0.05$). The AV from the Soxhlet method were higher than in the maceration method. According to FAO about the standard for fish oils (2017), the acceptable of AV on fish oil is ≤ 3 mg KOH/g. The high of AV can be caused by heat treatment while in the extraction process. The heat treatment cause oxidation and hydrolysis on PFO to produce free fatty acid (Herchi *et al*., 2016).

According to Table 2, the PCR technique gave the best result than others. The wavenumber of 2000-3100 cm^{-1} was chosen to determine AV because give the highest coefficient determination for calibration and validation, 0.9879 and 1.000 respectively with the lowest RMSEC and RMSEP were 0.210 and 0.972.

Figure 2 presents the linear regression of the PCR method expressing the relationship between AVs from the titration method (as actual value) and FTIR spectral (as calculated value). The peak at wavenumber of 2000-3100 cm^{-1} corresponding to the stretching vibration of cis -C=H at 3006 cm^{-1} and vibration of CH_2 stretching at the peak at 2854 and 2921 cm^{-1} from free fatty acid (Che Man *et al*., 2010) (Figure 3).

3.3 Determination of PV

Peroxide value (PV) is used to measure the level of peroxide/hydroperoxide formed at the initial process of oil oxidation by measuring iodine released from potassium iodide on titration process (Nunes, 2014; Naz and Saeed, 2018). The PVs of PFO by titration and FTIR combined with PCR method was shown in Table 1. Based on the ANOVA test, the PVs from titration and FTIR method were not significantly different ($p<0.05$). The accuracy of iodometric titration to determine the PVs depends on some other experimental factors such as precise timing and protection of the reaction mixture from oxygen (Armenta *et al*., 2007). Determination of PVS by titration method could have an error caused by

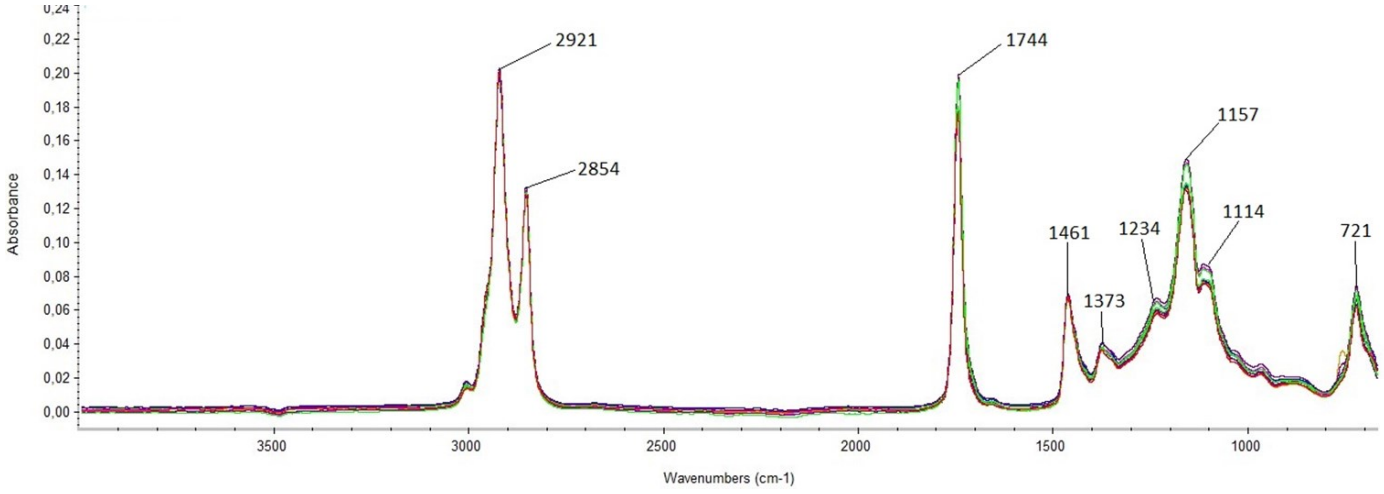

Figure 1. FTIR spectra of Patin fish oil from different extraction method at mid infrared region (4000-650 cm^{-1}) scanned with resolution of 16 cm^{-1} and number of scanning of 32.

Table 1. Determined of acid, peroxide, and saponification value by titration and FTIR methods after optimization method

Extraction methods	Solvent	Acid Value		Peroxide value		Saponification value	
		Titration	FTIR	Titration	FTIR	Titration	FTIR
Maceration methods (wet samples)	Hexane	5.27	5.42	9.59	9.48	135.21	142.48
	Chloroform	2.66	2.71	7.18	7.16	287.71	288.79
	Ethyl acetate	7.01	7.36	9.61	9.51	221.74	220.8
Maceration methods (dried samples)	Hexane	5.27	5.14	13.18	12.96	137.83	132.63
	Chloroform	8.86	8.88	13.18	12.97	287.64	284.41
	Ethyl acetate	10.57	10.84	13.16	13.5	187.84	189.93
Soxhlet	Hexane	15.6	15.67	8.38	8.52	144.28	145.44
	Chloroform	8.84	8.75	17.92	17.96	268.59	265.31
	Ethyl acetate	12.41	12.35	14.3	14.44	192.05	190.17

Table 2. Optimization of PCR and PLS methods to determine the acid value

Multivariate Calibration	Wavenumber (cm^{-1})	Spectra	Calibration		Prediction	
			RMSEC	R^2	RMSEP	R^2
PCR	653-826	Normal	1.78	0.8653	2.03	1.000
		1st derivative	3.56	0.0395	3.58	1.000
		2nd derivative	3.54	0.088	3.65	1.000
	1007-1600	Normal	1.96	0.8344	1.96	1.000
		1st derivative	2.55	0.6969	3.57	1.000
		2nd derivative	3.19	0.4421	4.03	1.000
	1500-2111	Normal	1.42	0.9171	2.33	1.000
		1st derivative	2.09	0.8088	0.361	1.000
		2nd derivative	2.07	0.8125	0.576	1.000
	2000-3100	**Normal**	**0.21**	**0.9983**	**0.972**	**1.000**
		1st derivative	0.6184	0.985	1.68	1.000
		2nd derivative	0.82	0.9731	1.5	1.000
	649-3005	Normal	1.12	0.9488	2.2	1.000
		1st derivative	1.5	0.9068	0.666	1.000
		2nd derivative	1.59	0.8952	0.377	1.000
PLS	653-826	Normal	3.56	0.0383	3.57	1.000
		1st derivative	3.56	0.0271	3.57	1.000
		2nd derivative	3.56	0.0272	3.57	1.000
	1007-1600	Normal	3.52	0.1491	3.97	1.000
		1st derivative	3.55	0.0744	3.78	1.000
		2nd derivative	3.55	0.0604	3.75	1.000
	1500-2111	Normal	1.92	0.8424	1.75	1.000
		1st derivative	3.46	0.2276	3.9	1.000
		2nd derivative	3.5	0.1801	3.91	1.000
	2000-3100	Normal	1.84	0.8564	2.04	1.000
		1st derivative	3.42	0.2778	4.07	1.000
		2nd derivative	3.45	0.2429	4.01	1.000
	649-3005	Normal	3.4	0.2979	3.67	1.000
		1st derivative	3.48	0.205	3.91	1.000
		2nd derivative	3.47	0.2196	4.09	1.000

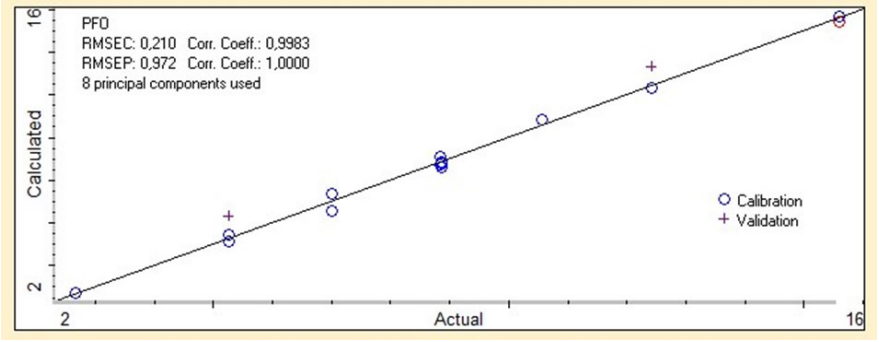

Figure 2. Plots of acid value result obtained by titration method (actual value) and FTIR method (calculated) in calibration and validation set using PCR technique

the mistake of distinguishing on the endpoint (Jiang *et al*., 2016). Peroxide/hydroperoxide which is intermediate species. These intermediates are unstable species that can react very quickly that make the mistake when the determination of endpoint (Mahboubifar *et al*., 2016). The acceptable of PV for fish oil is ≤ 5 meq O_2/kg oil (FAO, 2017). The oxidation rate increase with the temperature results high amount of peroxide, that make PVs of PFO from the Soxhlet method were highest than others (Popa *et al*., 2017).

Based on the optimization method, PLS with first derivative spectra showed the best result in the prediction of the desired response (Table 3). PLS at wavenumbers of 1500-2111 cm^{-1} were selected for determining the PVs because it gives the highest coefficient value for determination and validation. The coefficient value (R^2) of determination for the calibration and validation were obtained at 0.9982. The RMSEC and RMSEP value were 0.200 and 0.564 respectively. The excellent linear relationship between the PVs measured by titration and FTIR method shown in Figure 4. Figure 5 presents the area 1500 - 2111 cm^{-1} is attributed for -C=O ester stretching at wavenumber of 1700 cm^{-1} (Che Man *et al*., 2010). According to Guillén and Cabo (1999), under the oxidative conditions, the oils have a very intense absorption due to the ester carbonyl functional group of the triglycerides causes a peak at area 1746 cm^{-1}. They also found that secondary oxidation products that cause an absorption at 1728 cm^{-1} which overlaps with the peak of the ester functional group.

3.4 Determination of SV

Saponification value (SV) is an index of the average molecular mass of fatty acid in the oil sample (Zahir *et al*., 2017). The SV of PFO was determined by titration and FTIR combined with PCR and PLS methods were shown in Table 1. Both methods give not significantly different result ($p<0,05$). From the optimization method, the area at wavenumber of 1007-1600 cm^{-1} was chosen to determine PVs Table 4. The PLS method with first derivative spectra gave the best coefficient of determination for calibration and validation, 0.9970 and 1.000 respectively with the RMSEC and RMSEP were 4.54 and 4.45. This is supported by the result of research

Table 3. Optimization of PCR and PLS methods to determine the peroxide value

Multivariate Calibration	Wavenumber (cm^{-1})	Spectra	Calibration		Prediction	
			RMSEC	R^2	RMSEP	R^2
PCR	653-826	Normal	1.26	0.9256	1.85	0.8461
		1st derivative	1.41	0.9062	2.36	0.6824
		2nd derivative	1.41	0.9056	2.13	0.7450
	1007-1600	Normal	1.57	0.8829	2.09	0.7716
		1st derivative	1.57	0.8827	2.29	0.7091
		2nd derivative	1.32	0.9184	1.60	0.8695
	1500-2111	Normal	0.765	0.9733	0.599	0.9929
		1st derivative	1.91	0.8194	3.22	0.3592
		2nd derivative	1.47	0.8980	3.03	0.4763
	2000-3100	Normal	1.26	0.9263	1.21	0.9605
		1st derivative	1.08	0.9460	2.23	0.7201
		2nd derivative	0.825	0.9689	2.40	0.6906
	649-3005	Normal	0.998	0.9542	0.843	0.9869
		1st derivative	0.680	0.9790	0.330	0.9957
		2nd derivative	0.811	0.9700	0.642	0.9904
PLS	653-826	Normal	2.30	0.7255	2.82	0.7611
		1st derivative	2.02	0.7966	2.61	0.7397
		2nd derivative	2.13	0.7707	2.68	0.7493
	1007-1600	Normal	1.81	0.8391	3.47	0.2069
		1st derivative	1.64	0.8707	2.79	0.5305
		2nd derivative	1.65	0.8695	2.81	0.5314
	1500-2111	Normal	2.14	0.7680	1.88	0.8719
		1st derivative	**0.200**	**0.9982**	**0.564**	**0.9982**
		2nd derivative	0.683	0.9788	0.666	0.9871
	2000-3100	Normal	1.80	0.8411	3.31	0.3054
		1st derivative	1.77	0.8485	3.52	0.2450
		2nd derivative	1.81	0.8402	3.61	0.1963
	649-3005	Normal	2.25	0.7374	2.77	0.7514
		1st derivative	2.28	0.7297	2.82	0.7501
		2nd derivative	2.31	0.7211	2.86	0.7541

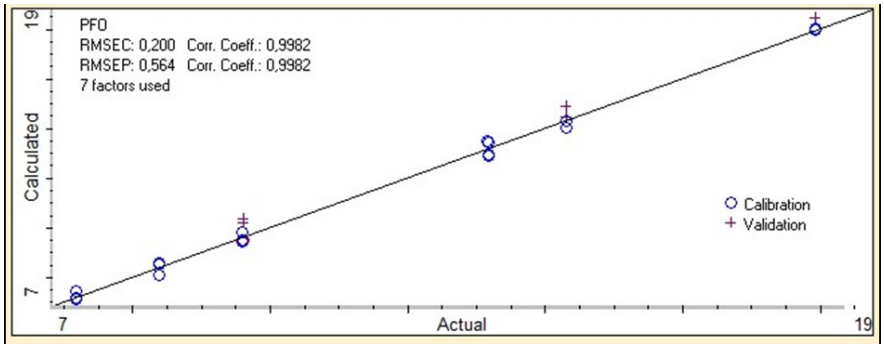

Figure 4. Plots of peroxide value result obtained by titration method (actual value) and FTIR method (calculated) in calibration and validation set using PLS technique

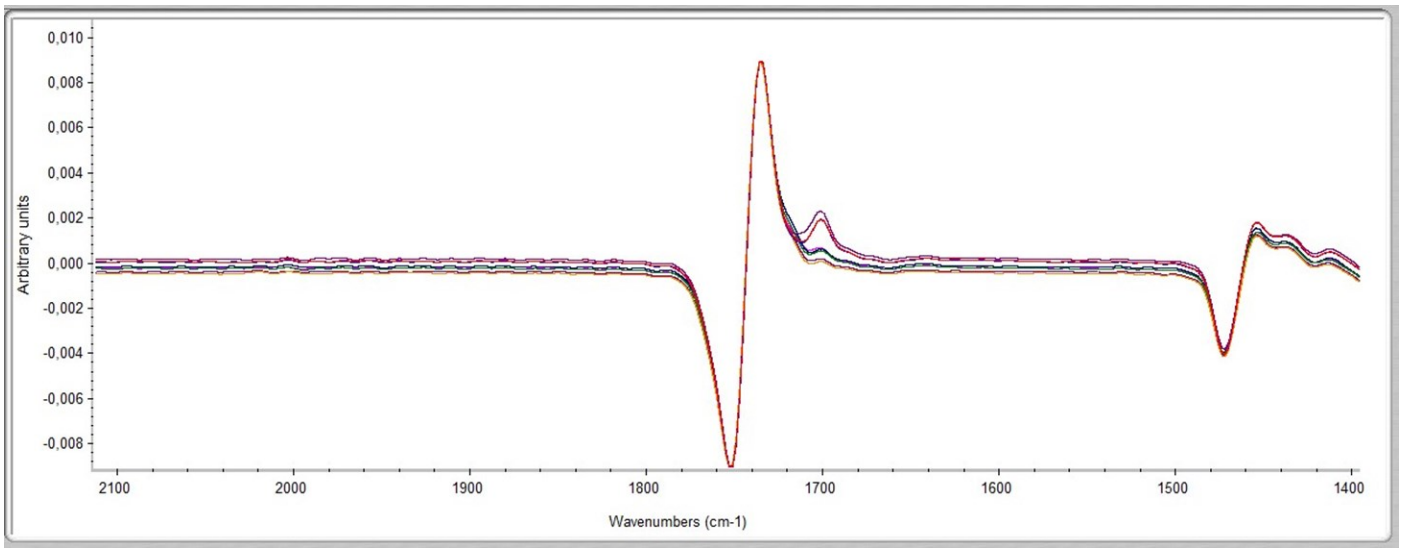

Figure 5. FTIR spectra to determine the peroxide value of Patin fish oil (1500-2111 cm^{-1})

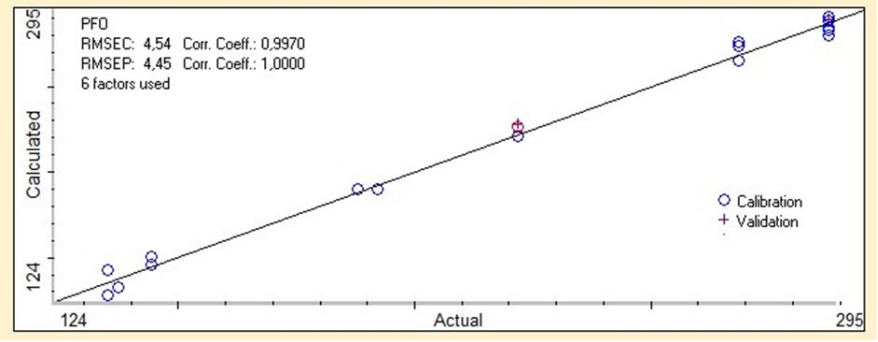

Figure 6. Plots of saponification value result obtained by titration method (actual value) and FTIR method (calculated)

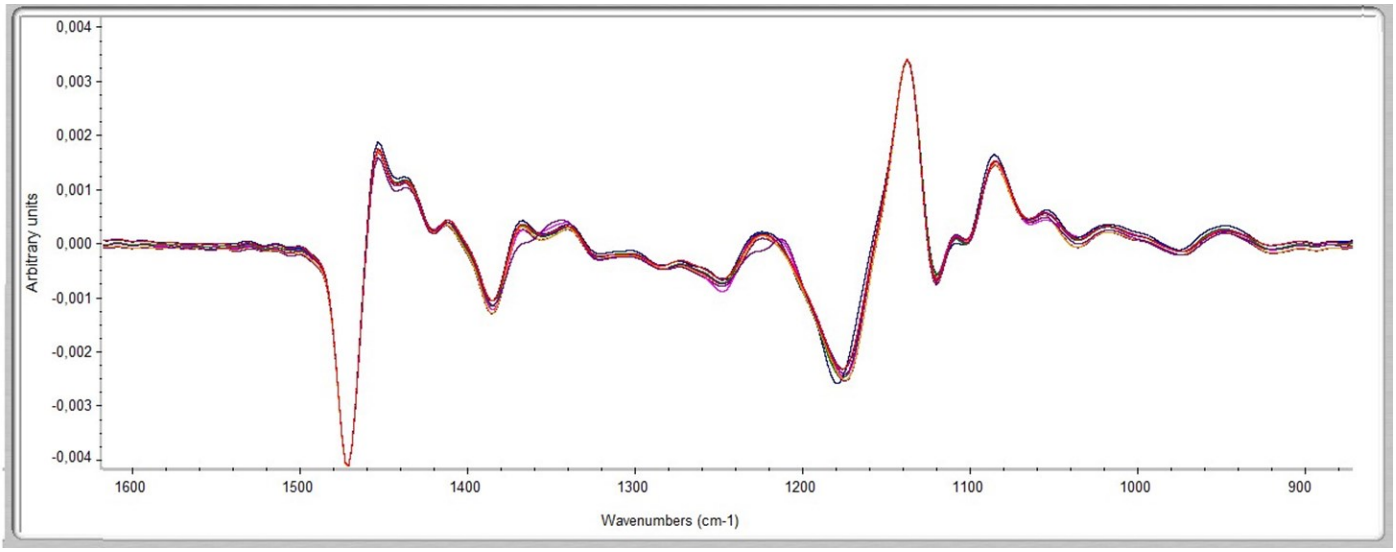

Figure 7. FTIR spectra to determine the saponification value of Patin fish oil (1007-1600 cm^{-1})

Table 4. Optimization of PCR and PLS methods to determine the saponification value

Multivariate Calibration	Wavenumber (cm^{-1})	Spectra	Calibration		Prediction	
			RMSEC	R^2	RMSEP	R^2
PCR	653-826	Normal	39.6	0.7412	57.6	1.000
		1st derivative	40.0	0.7337	57.0	1.000
		2nd derivative	41.3	0.7131	59.6	1.000
	1007-1600	Normal	14.9	0.9674	176	1.000
		1st derivative	19.2	0.9453	12.5	1.000
		2nd derivative	24.3	0.9112	18.4	1.000
	1500-2111	Normal	22.8	0.9221	46.3	1.000
		1st derivative	31.0	0.8507	53.6	1.000
		2nd derivative	26.6	0.8924	42.8	1.000
	2000-3100	Normal	9.14	0.9879	16.7	1.000
		1st derivative	18.3	0.9508	29.5	1.000
		2nd derivative	21.5	0.9310	30.9	1.000
	649-3005	Normal	8.17	0.9902	9.54	1.000
		1st derivative	9.84	0.9860	6.03	1.000
		2nd derivative	9.82	0.9860	4.11	1.000
PLS	653-826	Normal	44.8	0.6501	70.2	1.000
		1st derivative	44.6	0.6527	71.0	1.000
		2nd derivative	44.4	0.6569	71.2	1.000
	1007-1600	Normal	13.8	0.9721	15.2	1.000
		1st derivative	**4.54**	**0.9970**	**4.45**	**1.000**
		2nd derivative	18.2	0.95113	10.3	1.000
	1500-2111	Normal	21.3	0.9321	45.3	1.000
		1st derivative	36.8	0.7811	67.4	1.000
		2nd derivative	35.5	0.7983	64.0	1.000
	2000-3100	Normal	8.96	0.9884	15.6	1.000
		1st derivative	16.4	0.9606	27.2	1.000
		2nd derivative	17.1	0.9571	25.8	1.000
	649-3005	Normal	45.1	0.6444	70.2	1.000
		1st derivative	45.0	0.6462	70.8	1.000
		2nd derivative	45.0	0.6462	70.8	1.000

conducted by Rohman *et al*. (2015) that use absorbance at wavelength of 1400-1600 cm^{-1} with PLS technique to determine the SVs. The linear regression as shown in Figure 6. First derivative spectra were displayed in Figure 7.

4. Conclusion

In this work, the AV, PV, and SV of PFO were successfully determined via FTIR spectroscopy combined with chemometrics using PCR and PLS method. The rapid determination of AV, PV, and SV by FTIR spectroscopy is, therefore, suitable and practical option for process control. Another advantage of FTIR spectroscopy method is that it is environmentally friendly as no chemical is needed except acetone for cleaning the ATR crystal. By utilizing this method, the chemical cost is negligible as compared to the AOCS standard method.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgment

The author thanks to The Ministry of Research, Technology, and Higher Education of the Republic of Indonesia for its scholarship so that this research can be done.

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eISSN: 2550-2166