

Research Article

Feasibility Study on the Use of a Portable NIR Spectrometer and Multivariate Data Analysis to Discriminate and Quantify Adulteration in Fertilizer

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The rise in population growth worldwide requires efficient management of agricultural lands through the correct determination of authentic fertilizers. In this current study, a rapid on-site detection technique was developed by using portable NIR spectroscopy in the wavelength range of 740–1070 nm together with optimum multivariate algorithms to identify fertilizer integrity (unexpired, expired, and adulterated) as well as quantify the levels (10–50%) of adulteration. NIR models were built based on support vector machine (SVM) and random forest (RF) for identification, while different types of partial least square regression (PLS, iPLS, Si-PLS, and GaPLS) were used for quantification purposes. The models were evaluated according to identification rate (Rt), coefficient of correlation in prediction (Rpre²), and root mean square error of prediction (RMSEP). For the identification of the integrity of the fertilizer, among the mathematical pretreatments used, the first derivative (FD) together with SVM gave above 99.20% identification rate in both calibration and prediction sets. For the quantification of the adulterants, Si-PLS was found to be superior and showed an excellent predictive potential of Rpre² = 0.95–0.98 and RMSEP = 0.069–0.11 for the two fertilizer types used. The overall results indicated that a handheld NIR spectrometer together with appropriate algorithms could be employed for fast and on-site determination of fertilizer integrity.

1. Introduction

The rise in population growth worldwide puts a lot of pressure on agricultural resources. This rapid growth has called for agricultural intensification to supply the necessities of life, which include healthy food. To address the demand for food to support the rising population growth, agro-inputs such as fertilizers are rigorously used in agriculture to increase productivity [1]. Recent studies indicated that fertilizer use in agriculture has resulted in [1, 2]. Nevertheless, the proliferation of fertilizer has created an opportunity for fraudsters to cheat by either selling fake, adulterated, or expired fertilizer, and often this mischief goes unnoticed and the farmers are at the receiving end. Farmers intend to also pass on their associated burden to consumers. therefore, time for policymakers and major players in the agriculture value chain to have a rapid detection technique to assist in the precision measurement of fertilizer integrity to help regulators ensure its quality and protect the brand. This would promote high productivity and traceability to guarantee farmers' commitment to safe food production [3] and contribute to sustainable development goals. The application of portable NIR spectroscopy coupled with a systematic selection of a multivariate algorithm for differentiating expired and unexpired chemical fertilizers would be very useful.

Global food demand will continue to grow in the coming decades, with concomitant challenges for sustainable agricultural input supply. The increase in agricultural productivity is strongly linked to the intensive use of fertilizer [4, 5]. However, the misuse, sale, and proliferation of expired, adulterated, and fake fertilizers are known to lead to poor germination, weak soil nutrients, water and air pollution, and serious problems for human health and the environment at large [6, 7]. To attain maximum agricultural output, there is an urgent need for the cautious use of natural resources and minimal application of hazardous compounds [1]. To make matters more complicated, the sale and use of banned, fake, and counterfeit fertilizers in developing countries are perceived. This act is often either not researched or not reported in the literature. This calls for immediate research attention to curb this menace. The solution lies in the development of rapid and farmer-friendly detection technology coupled with a smartphone device. For instance, in Ghana, the Environmental Protection Agency (EPA) and Ghana Standards Authority (GSA) are not able to do due diligence in monitoring, controlling, and managing fake or adulterated agro-inputs for the production of major staple foods. This is due to the cumbersome, expensive, and slow nature of the analytical techniques available.

Until now, there are available rapid on-site and nondestructive measurement techniques for the determination of fertilizer integrity. There is therefore a need for the development of rapid detection techniques that are appropriate for our geographical location. The application of portable spectrometers and chemometrics coupled with smartphone technology holds more promise. Spectroscopy has been used for measuring other attributes in commodities like cocoa [8, 9], seed germination [10, 11], and soil health [12, 13]. However, little or no research has reported on the feasibility of portable NIR spectroscopy for measuring authentic, adulterated, and expired fertilizer.

2. Materials and Methods

In this study, two brands of fertilizer samples were used, as shown in Table 1. The first category was brand A (authentic improved urea, expired improved urea, and adulterated improved urea) and brand B (authentic nitrabor, expired nitrabor, and adulterated nitrabor). The formulation of the adulterated category was done by spiking the two authentic fertilizers differently with various levels of expired ones in the range of 10–50 wt/wt.%. The authentic samples were obtained from registered and recommended fertilizer dealers in Cape Coast, Kumasi, and Accra, while their expired counterparts (expired 1.5–3 years) were also obtained from the Department of Soil Science at the University of Cape Coast. All samples were labelled and transported into the laboratory for further analysis.

2.1. Spectral Acquisition. The spectrum of each unique sample was taken in the reflectance mode using a handheld spectrometer (SCIO^R) in the wavelength range of 740-1070 nm with a 1-nm resolution for spectra data readings operated with the aid of Scio Lab assisted by a smartphone device. The SCIO (Model CP-SCM001) portable NIR used in this study is from consumer physics; it weighs $35 g (68 \times by 40.2 \times 18.8 \text{ mm})$ and employs Bluetooth to connect to a smartphone or tablet (Android or iOS) and an SCIO lab app, which operates using Internet connectivity for a two-way communication when scanning. For more information, refer to other authors [14, 15]. Each fertilizer sample was collected into a sample cup and scanned three times after rotating the sample 45°. The whole process was carried out at a temperature of $31 \pm 2^{\circ}C$ and a relative humidity of $60 \pm 3\%$. The spectra data stored at the cloud base was downloaded and transported into MATLAB (R2020a; The MathWorks, Natick, MA), where all the calculations and modelling were done in Windows 10. The reflectance spectra were then transformed into absorbance by using

$$A = Log10\frac{1}{R}.$$
 (1)

2.1.1. Chemometric Techniques. All the chemometric techniques used in this experiment for spectra data analysis included preprocessing tools such as Savitzky-Golay first derivative (FD), Savitzky-Golay second derivative (SD), multiplicative scatter correction (MSC), and standard normal variant (SNV). These aforementioned preprocessing techniques were done to comparatively evaluate their known unique impacts, such as removing baseline drifts, multiplicative scattering effects, background noise, and signal noise, as proposed by other authors [16, 17]. After processing the spectra data set, identification models (random forest; RF; and support vector machine; SVM) and quantification models (partial least square, PLS; interval partial least square, iPLS; synergy interval partial least square, Si-PLS; and back interval partial least square, Bi-PLS) were built for identifying authentic from expired ones as well as quantifying the adulterant in the pure samples. For more information on the theories of the identification and quantification models used, refer to other authors [8, 18, 19]. The optimal identification and quantification models' performances were compared among the different algorithms using equations (2)-(7), respectively. More specifically, the identification rate would be evaluated using the percentage accuracy in the prediction set, while it is mostly a rule to judge the performance of the quantification model using statistical parameters such as the correlation coefficient in the prediction set and the root mean square error in the prediction set.

No.	Categories	Fertilizer type	No. of samples	Percentage purity (%)	Chemical	Physical
	Authentic = A	Improved urea	63	100	40% N+5.6% S	Nitrovan ailmhata ailmhiir amaiilar fartilizar that ia 10006 adiibha
	Expired = A	Improved urea	63	100	40% N+5.6% S	murugen suipnate suipnu granuar terunzer unat 18 100% sonuore
			35	10		
Brand A			35	20		
	Adulterated = A	Improved urea	35	30	Nil	Pure improved urea samples adulterated with expired ones
			35	40		
			35	50		
	Authentic $= B$	Nitrabor	63	100	15.4% N+25.9% CaO+0.3% B	Dourse environ field and de solations without foundiirous
	Expired = B	Nitrabor	63	100	15.4% N+25.9% CaO+0.3% B	Delise granmar menu grade calcium mutate termizer
			35	10		
Brand B			35	20		
	Adulterated = B	Nitrabor	35	30	Nil	Pure nitrabor samples adulterated with expired ones
			35	40		
			35	50		
	Total s	amples		602		

TABLE 1: Samples used for the experiment.

Accuracy(%) =
$$\frac{TN + TP}{Total} \times 100,$$
 (2)

Sensitivity (%) =
$$\frac{TP}{Tp + FN} x100,$$
 (3)

Specificity (%) =
$$\frac{TN}{TN + FP} x100,$$
 (4)

$$\operatorname{Error rate}(\%) = \frac{FP + FN}{Total} \times 100,$$
(5)

$$Rpre = \sqrt{1 - \frac{\sum_{i=1}^{n} \left(\hat{y}_{i} - y_{i}\right)^{2}}{\sum_{i=1}^{n} \left(y_{i} - \overline{y}\right)^{2}}},$$
(6)

$$RMSEP = \sqrt{\frac{\sum_{i}^{n} (y_{i} - \hat{y}_{i})^{2}}{n}}.$$
(7)

3. Results and Discussion

3.1. Spectra Examination. In this feasibility study, two categories of fertilizers were used separately and simultaneously, namely brand A (authentic improved urea, expired improved urea, and adulterated improved urea) and brand B (authentic nitrabor, expired nitrabor, and adulterated nitrabor) fertilizers (improved Urea and nitrabor) were investigated using portable NIR spectroscopy. As seen in Figure 1, the raw spectra profile and Savitzky-Golay second derivative (SGSD) spectra profile reveal useful peaks associated with ROH, RNH, CH, CH₂, and CH₃ hydrogen bonds at the third overtone region [20]. For the raw spectra as seen in Figure 1(a), a major peak was observed at 950 nm to 1000 nm, while it was observed that other peaks were magnified when the second derivative technique was used, as seen in Figure 1(b). These peaks are located around 780 nm, 830 nm, 940 nm, 975 nm, and 1025 nm, and these peaks could be associated with RNH₂, CH, and ArCH [21] in fertilizer A (improved urea), which contains sulphur-enriched granular ureabased fertilizer with different levels of nitrogen and sulphur oxide (40% N and 14% SO₃). In Figure 2, it was seen that fertilizer brand B (nitrabor) had two major peaks for raw spectra around 800 nm and 1025 nm, which are located in the third overtone region. While for the SGSD spectra profile (Figure 2(b)), the major peaks were observed at several wavelengths, including 775 nm, 850 nm, 900 nm, 1000 nm, and 1045 nm. These wavelengths correspond to the third overtone region associated with CH₃, CH₂, CH, RNHR, and RNH₂ [20, 22]. These compounds could be related to the chemical properties of the fertilizer samples used, as it has been observed that spectral fingerprints normally originate from a combination or overtones of C-H stretching modes of saturated CH₂ and CH₃, or aromatic functional groups [23, 24].

3.2. Principal Component Analysis (PCA). PCA of the various categories was observed after different applications of preprocessing treatment were compared with each other. It was observed for fertilizer A that both the raw spectra and the other two techniques (SNV and SGFD) could not give a clear separation of the three categories, as seen in Figures 3(a)-3(c). However, the SGSD PCA cluster plot showed a well-defined separation of the three distinct fertilizer categories used, as seen in Figure 3(d). More so, this same phenomenon was observed by fertilizer B, as seen in Figures 4(a)-4(c). This means that SGSD spectra preprocessing treatment techniques work better than the others for clustering fertilizer categories. Also, it could be observed that fresh fertilizer is different from expired and adulterated fertilizers. These separations could be attributed to differences in the chemical properties of the categories of fertilizer. Most importantly, the expired fertilizers could have lost their potency because fertilizers stored poorly under unfavourable temperature and humidity conditions can lose their rich chemical properties and break down their nutrients. Under prolonged storage under high-temperature conditions, nitrogen is lost [25-27]. It is also believed that fertilizer does not break down easily but can, however, change its chemical properties by chemical reaction or become free ions at high humidity and moisture as ammonia volatilization is the main mechanism of nitrogen loss from urea [28, 29].

4. Identification Model

Furthermore, identification models were comparatively built and evaluated. These models included random forest (RF) and support vector machine (SVM). The models revealed optimum results after preprocessing treatments such as SNV, FD, and SD. From Table 2, it can be seen that all the identification models performed well above a 90% identification rate. However, after preprocessing the raw spectra data set, the second derivative spectra preprocessing treatment performed comparatively better than the others, with a 97% identification rate when RF was employed, while when SVM was also used, it gave a 99% identification rate. The best performance by SVM could be attributed to the potential of the model overcoming overfitting and the strong nonlinear supervised attribute, as well as the higher generalization of SVM by maximizing the margin and supporting efficient learning of nonlinear functions [30]. Furthermore, the major peaks that contributed to the neat clustering and strong identification rate are described by the Eigenvectors value for the identification of fertilizer categories, as seen in Figure 5. From this Figure, it can be seen that the major peaks are 800 nm, 925 nm, and 975 nm, which correspond to CH, CH₂, and CH_3 in the third overtone region [20].

For fertilizer B, the identification rate observed was between 93% and 97% for the RF with different preprocessing treatments. A similar trend was observed for



FIGURE 1: Spectra profile: (a) raw and (b) SGSD of fertilizer A categories.



FIGURE 2: Spectra profile: (a) raw and (b) SGSD of fertilizer B categories.

fertilizer B, as the second derivative spectra performed better than the other preprocessing treatments. On the other hand, as seen in Table 3, SVM also gave a superior identification rate than the RF algorithm. This phenomenon further proved that SVM shows good performance for classifying high-dimensional data when a limited number of samples are available. Hence, it has increasingly become a very popular classification algorithm with strong nonlinear supervised potential [31, 32]. From Figure 6, it can be seen that the major wavelengths that contributed to the accurate identification are centered around 800 nm, 960 nm, 1025 nm, and 1040 nm. These wavelengths are associated with the second overtone region, which represents nitrogen.

5. Quantification Model

In this research, an attempt to quantify the adulteration of the expired fertilizer was made for the first time using various partial least square multivariate algorithms. From the study, it was observed that the NIR spectra data set could be a model for rapid quantification of fertilizer integrity. For fertilizer A, as seen in Table 4, it could be observed that all the quantification models developed had prediction rates above 0.93 (R^2). These results proved the feasibility of rapid determination of fertilizer integrity to aid accurate agricultural production and increase yield. Furthermore, it must be emphasized that among the models developed, the Si-PLS



FIGURE 3: PCA score plot: (a) raw, (b) SNV, (c) SGFD, and (d) SGSD of fertilizer brand A.

model performed superior to the others with a coefficient of determination of $R^2 = 0.94$ and 0.95 in the training and test sets, respectively, with the selected wavelengths of 820–839 nm, 938–956 nm, and 995–1013 nm, as seen in Figure 7. While its closest model Bi-PLS also selected similar wavelengths of 825–841 nm, 927–942 nm, and 959–975 nm. These wave bands in the spectra wavelength region used in this study were those responsible for the correct identification of the categories of samples used [33].

More so for fertilizer B, the quantification of the adulteration was successfully achieved above $R^2 = 0.95$, as seen in Table 5. This means that each of the developed models could be used for industrial and market surveillance analyses of fertilizer integrity. However, for improved performance, the Si-PLS model used its superiority over the others. The optimum wavelength ranges selected for the superior model were 740–767 nm, 824–851 nm, and 990–1016 nm, as seen in Figure 8. This means that the selected wavelengths correlated well for the quantification of adulteration of the fertilizer used in this experiment. The wavelengths are also associated with third overtone regions with functional groups such as NH₂, C-H, and RNH₂. These groups, especially NH₂ groups, are known to vanish as chemicals expire [34].

6. Simultaneous Measurement of Adulteration Levels in Fertilizer Brands A and B

After a successful classification and quantification of individual fertilizers (A and B), this research further exploited the feasibility of quantifying adulteration levels in two



FIGURE 4: PCA score plot: (a) raw, (b) SNV, (c) SGFD, and (d) SGSD of fertilizer brand B.

		Preprocessing treatment at 5PCs							
Models	Evaluation	Raw		SN	SNV		D	S	D
		Train	Test	Train	Test	Train	Test	Train	Test
	Accuracy	93.70	94.70	93.50	94.60	96.50	96.50	93.70	94.70
RF	Error rate	6.30	5.30	6.50	5.40	3.50	3.50	6.30	5.30
	Sensitivity	96.40	96.00	95.30	94.00	96.80	97.60	96.40	96.00
	Specificity	99.70	99.70	98.90	99.50	98.50	99.20	99.70	99.70
CVIM	Accuracy	92.30	92.30	98.50	98.50	99.20	99.50	92.30	92.30
	Error rate	7.70	7.70	1.50	1.50	0.80	0.50	7.70	7.70
5 V IVI	Sensitivity	96.30	97.10	98.50	96.80	99.20	99.50	96.30	97.10
	Specificity	92.30	92.30	97.50	97.50	99.20	99.50	92.30	92.30

TABLE 2: The influence of preprocessing technique on identification models (fertilizer A).



FIGURE 5: Eigenvectors for the identification of fertilizer brand A category.



FIGURE 6: Eigenvectors for the identification of fertilizer brand B category.

TABLE 3: The influence of preprocessing technique on identification models (fertilizer B).

		Preprocessing treatment at 5 PCs							
Models	Evaluation	Raw		SI	SNV		FD		D
		Train	Test	Train	Test	Train	Test	Train	Test
RF	Accuracy	97.50	97.50	97.40	97.40	97.40	97.50	98.00	98.00
	Error rate	2.50	2.50	2.60	2.60	2.60	2.50	2.00	2.00
	Sensitivity	98.10	98.40	98.00	97.50	92.10	91.50	98.00	98.00
	Specificity	99.60	99.60	99.20	99.20	99.60	99.60	99.60	99.60
	Accuracy	98.70	98.70	99.30	99.40	99.50	99.50	99.70	99.40
	Error rate	1.30	1.30	0.70	0.60	0.50	0.50	0.30	0.60
SVM	Sensitivity	98.20	99.50	99.70	99.80	99.50	99.50	99.40	99.60
	Specificity	99.70	99.70	98.60	97.90	97.60	96.80	98.20	97.80

Model	R^2	RMSECV	Bias	R^2	RMSEP	Bias	RPD	
PLS	0.9351	0.1297	0.0094	0.9450	0.1201	0.0133	3.0767	
iPLS	0.9346	0.1302	-0.0003	0.9433	0.1227	-0.0045	3.0128	
Bi-PLS	0.9424	0.1224	-0.0005	0.9299	0.1362	-0.0134	2.7141	
Si-PLS	0.9496	0.1147	0.0004	0.9529	0.1122	-0.0076	3.2947	
GAPLS	0.9409	0.1240	-0.0011	0.9493	0.1157	0.0010	3.1950	
	Variables					d wavelength range	e (nm)	
PLS	LS 331					740-1070		
iPLS	S 16				927-942			
Bi-PLS	50				825-841, 927-942, 959-975			
Si-PLS				820-839, 938-956, 995-1013				
GAPLS			331	740-1070				

TABLE 4: Optimum performance of different quantification models for fertilizer A.



FIGURE 7: Si-PLS spectra selection: (a) and model plots (b) for NIR estimation and measured value.

Model	R^2	RMSECV Bias R			RMSEP	RMSEP Bias		
PLS	0.9523	0.0801	0.0054	0.9529	0.0898 0.0092 4.344			
iPLS	0.9613	0.0945	0.0004	0.9648	0.0908	-0.0007	3.8200	
Bi-PLS	0.9664	0.0881	0.0881 -0.0002 0.9676			-0.0050	3.9460	
Si-PLS	0.9767	767 0.0736 0.0007 0.97			0.0692	-0.0012	5.0124	
GAPLS	0.9736	0.0782	0.0004	0.9766	0.0742	0.0028	4.6746	
	Variables				Selecte	d wavelength range	e (nm)	
PLS	331					740-1070		
iPLS	17				757–773			
Bi-PLS		47			757-773, 959-974, 1009-1022			
Si-PLS			83		740-767, 824-851, 990-1016			
GAPLS				740-1070				

TABLE 5: Optimum performance of different quantification models for fertilizer B.

fertilizer groups simultaneously. It was observed that the NIR spectra data set could be trained to quantify adulteration levels in different types of fertilizers. The models used obtained results between 0.81–0.94 and 0.79–0.92 of R^2 in the training set and test set, respectively, as seen in Table 6. It was observed that the PLS model had the least performance ($R^2 = 0.81$ and 0.79 using variables 331), while the best performance was obtained for Si-PLS at 61 variables with $R^2 = 0.94$ and 0.92 in the training set and test set, respectively.

It could be explained that Si-PLS used 61 variables and made optimum use of these selected wavelength ranges of 740–755 nm, 816–830 nm, 936–950 nm, and 966–980 nm that corresponded accurately with the group of fertilizers



FIGURE 8: Si-PLS spectra selection: (a) and model plots (b) for NIR estimation and measured value.

Model	R^2	RMSECV	Bias	R^2	RMSEP	Bias	RPD	
PLS	0.8133	0.2064	0.0102	0.7966	0.2139	0.0161	1.6589	
iPLS	0.8920	0.1603	-0.0003	0.8824	0.1692	-0.0198	2.0972	
Bi-PLS	0.9156	0.1426	0.0002	0.7068	0.2537	-0.0366	1.3987	
Si-PLS	0.9460	0.1150	-0.0003	0.9295	0.1328	-0.0193	2.6720	
GAPLS	0.8895	0.1620	-0.0002	0.8551	0.1864	-0.0135	1.9020	
				Selecte	d wavelength range	e (nm)		
PLS	S 331					740-1070		
iPLS	16				959–974			
Bi-PLS	50				740-756, 808-824, 927-942			
Si-PLS			61		740-755, 816-830, 936-950, 966-980			
GAPLS			331		740-1070			





FIGURE 9: Si-PLS spectra selection (a) and plot (b) of NIR estimation and measured value.

used and their levels of adulteration as observed in Figure 9. Furthermore, Si-PLS is known to possess the potential of selecting multiple spectra subintervals that are informative enough to provide optimum results as well as eliminate irrelevant information that could influence the performance of the model [35].

7. Conclusion

This research has shown that on-site and rapid detection of fertilizer integrity is feasible and could be an effective tool for efficient quality control of fertilizer. In this study, the first derivative (FD) preprocessing together with either SVM gave above 99.20% identification rate in both the calibration and prediction sets. For the quantification of the adulterants, Si-PLS was found to be superior and showed an excellent $Rpre^2 = 0.95 - 0.98$ predictive potential of and RMSEP = 0.069 - 0.11 for the two fertilizers used. The results only showed feasibility studies, and further studies are needed to include a wide range of samples and potential adulterants to improve the robustness of the models. It could offer a user-friendly technique for monitoring and quality control services in the fertilizer value chain.

Data Availability

The data supporting the findings of this study will be made available upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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