DETECTION OF SLUDGE CONTAMINATION IN CRUDE PALM OIL USING DIELECTRIC SPECTROSCOPY

K. Hamdan, S. Abd Aziz, A. Yahya, F. Z. Rokhani, B. L. Steward

ABSTRACT. *Degradation and deterioration of crude palm oil (CPO) is a major concern in the palm oil industry because it could affect the quality of palm oil products. The development of rapid and non-destructive measuring techniques is needed to enhance the efficiency of palm oil quality monitoring. This study used dielectric spectroscopy to detect possible sludge contamination in CPO. Dielectric properties of CPO were measured at different temperatures and sludge contamination levels using a liquid dielectric test fixture that was connected to an impedance analyzer with frequencies ranging from 3 to 30 MHz. The variations in dielectric properties were analyzed using ANOVA and Duncan's multiple range test. Principal component regression (PCR) and partial least squares (PLS) analysis were used for model development to predict sludge contamination. The results showed that there was a significant difference in dielectric constant as the temperature increased from 28°C to 55°C ($p < 0.0001$). The dielectric constant also increased from 3.01 to 63.53 with increasing contamination levels. Generally, there were significant differences between the dielectric constants of pure and contaminated CPO ($p < 0.0001$). The PCR and PLS calibration models showed good prediction ability of sludge contamination at different temperature levels. The classification of sludge contamination yielded very strong correlation, with r^2 values ranging from 0.91 to 0.98. The best result was obtained at 28°C with the lowest value of standard error cross-validation (SECV) of 1.04%. The results showed that dielectric spectroscopy has strong potential for CPO quality monitoring.*

Keywords. *Crude palm oil, Dielectric spectroscopy, Quality monitoring, Sludge contamination.*

Deterioration and degradation of crude palm oil (CPO) is a major concern in the palm oil industry. Depending on the processing method, CPO could deteriorate at several stages of the palm oil milling process, which consequently affects its quality. For example, contamination and degradation might occur due to sterilizer condensate formation during sterilization, unused heat and steam injection during the fruit digestion stage, high pressures used for separating poisonous hydraulic fluid and oil during the pressing stage, badly oxidized sludge oil during clarification, and overheated CPO in the storage tank after purification (Gee, 1999).

Perumal (2009) reported that CPO contamination also occurred after the milling stage due to illegal siphoning activities while the CPO was being transported from the mill to refineries. The amount siphoned off by syndicates was replaced by other liquids, such as sludge or used oil, before the CPO arrived at the refineries. A total of 39 cases of siphoning activities were reported in 2010, which involved 252.73 tons of CPO worth USD $201,766 (Bernama, 2011). In a recent case, an Indonesian palm oil company was blamed for the diesel oil contamination of 19,000 metric tons of CPO exported to Rotterdam, Netherlands. The incident caused the Netherlands to suspend some import contracts and tarnished the image of Indonesian exporters in the Netherlands (Jakarta Post, 2009). As CPO contamination is a major concern in the palm oil industry, it is important to have an efficient method for CPO monitoring. Generally, a number of methods are used to monitor and determine CPO quality. Parameters such as impurities and moisture content, free fatty acids (FFA), the deterioration of bleachability index (DOBI), iodine value, and peroxide value are commonly measured using laboratory tests such as wet chemical analyses and gas liquid chromatography (Ainie et al., 2005). The procedures used to determine these parameters are laborious, time-consuming, and require skilled operators. To solve this problem, Moh et al. (1999a) developed a near-infrared (NIR) spectroscopy method to measure peroxide value in CPO. Che Man and Moh (1998) also investigated NIR spectroscopic techniques for the determination of FFA in CPO. Their study showed that NIR spectroscopy can reduce the time required for sample analysis when compared to conventional wet chemical analysis. The amount of hazardous solvents as well as the cost of labor can also be reduced. More recently, Fourier transform infrared (FTIR) spectroscopy has been used to estimate FFA, moisture content, peroxide value, and iodine value of CPO, as well as in the analysis of the extra virgin

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The authors are **Khairunnisa Hamdan,** Graduate Student, **Samsuzana Abd Aziz, ASABE Member,** Senior Lecturer, and **Azmi Yahya, ASABE Member,** Associate Professor, Department of Biological and Agricultural Engineering, Universiti Putra Malaysia, Serdang, Malaysia; **Fakhrul Zaman Rokhani,** Senior Lecturer, Department of Computer and Communication Systems Engineering, Universiti Putra Malaysia, Serdang, Malaysia; **Brian L. Steward, ASABE Member,** Professor, Department of Agricultural and Biosystems Engineering, Iowa State University, Ames, Iowa. **Corresponding author:** Samsuzana Abd Aziz, Department of Biological and Agricultural Engineering, Faculty of Engineering, 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia; phone: +603-8946-4455; e-mail: samsuzana@upm.edu.my.

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olive oil adulterated with palm oil (Che Man et al., 1999, 2000; Moh et al., 1999b; Rohman and Che Man, 2010).

Recently, dielectric spectroscopy has been applied to the analysis and monitoring of agricultural and food product quality (Nelson, 2005; Cataldo et al., 2009; Nelson et al., 2005, 2007; Bodakian and Hart, 2002) and in oil-related industrial operations (Dongzhi, et al., 2008). The potential of using dielectric spectroscopy in vegetable oil quality monitoring has been studied and reported. Lizhi et al. (2008) conducted a study on the dielectric properties of edible oils and fatty acids as a function of frequency, temperature, moisture, and composition. The results indicated that the dielectric constant of corn oil increased with increasing moisture content and decreased significantly with increasing frequency. Their study also showed a positive result in discriminating adulterated edible oils. They concluded that information on dielectric properties could be useful in oil identification, quality evaluation, and quality monitoring during oil processing and storage. Lizhi et al. (2010) conducted a further study to distinguish olive oil adulterated with other vegetable oils. Their study showed good prediction capability for different concentrations of adulterants in olive oil. Cataldo et al. (2009) investigated the dielectric properties of several vegetable oils, such as peanut, corn, sunflower, soybean, olive, and various seed oils, using microwave dielectric spectroscopy. They found that the relaxation frequency differed among the vegetable oils, and it was a key to identifying different kinds of oil. They suggested that dielectric spectroscopy has potential for practical application in adulteration detection and oil quality estimation.

Based on the results from this previous relevant research, there is great potential for using dielectric spectroscopy in monitoring oil quality. In this research, dielectric spectroscopy was used to investigate the variation of dielectric spectra of CPO and to detect sludge contamination in CPO. Specifically, the objective of this work was to investigate the effect of sludge contamination on the dielectric properties of CPO at various temperatures with different contamination levels and measurement frequencies. Multivariate data analysis was conducted to develop prediction models to estimate the amount of sludge contamination in CPO. The results showed that dielectric spectroscopy is useful for sludge contamination detection in CPO.

Material and Methods

Sample Preparation

The CPO samples were obtained from a local palm oil mill in Banting, Malaysia. The oil samples were taken from the storage tank, which is the last storage of CPO before it is transported to the refineries. The CPO used in this study contained 0.025% moisture, 4.50 FFA, and 0.01% dirt (impurity). A palm oil sludge sample was obtained from the clarifying station of the same processing mill. Sludge oil refers to the brown effluent slurry that is generated in the clarification process when the pure oil and sludge are separated. It was composed of 4% to 5% solids, mainly organic, 0.5% to 1% residual oil, and about 95% water. The CPO samples were then artificially contaminated by the addition of sludge oil at four contamination levels: 0.6%, 1.0%, 5.0%, and 10.0%.

Instrumentation Setup and Dielectric Properties Measurement

Dielectric property measurement of CPO was performed using an Agilent liquid dielectric test fixture (model 16452A, Agilent Technologies, Hyogo, Japan) connected to an Agilent precision impedance analyzer (model 4294A, Agilent Technologies, Hyogo, Japan) at frequencies ranging from 3 to 30 MHz with 100 Hz intervals.

Dielectric measurements were acquired at four temperature levels (28°C, 35°C, 45°C, and 55°C) within the range of conditions during CPO transport. The sample temperature was monitored using a temperature probe that was inserted into the CPO sample and connected to a programmable stirring hot plate. The stirring hot plate was used to heat the sample to the desired temperature. The oil sample was circulated into the test fixture using a digital peristaltic pump (fig. 1).

The air capacitance (C_0) of the test fixture was obtained at room temperature after the measurement system was calibrated (Agilent, 2000). The dielectric properties of the oil sample at different sludge contamination and temperature levels were then measured across the frequency range. After each sample measurement, the fixture was disassembled, cleaned, and dried at room temperature. The test was replicated three times with a randomized order of temperature and contamination levels.

The dielectric constants of the pure and contaminated CPO samples were calculated using the following equation:

$$\varepsilon' = \alpha\left(\frac{C_p}{C_0}\right) \tag{1}$$

where ε' is the dielectric constant, α is the corrective coefficient, and Cp is the oil capacitance (pF). The corrective coefficient (α) was calculated using the following equation (Agilent, 2000):

$$\alpha = \frac{100\left|\dot{\varepsilon}_{rm}\right|}{97.0442\left|\dot{\varepsilon}_{rm}\right| + 2.9558} \tag{2}$$

where $$\left|\dot{\varepsilon}_{rm}\right| = \sqrt{\frac{{C_p}^2}{{C_0}^2} + \frac{1}{(\omega C_0 R_p)^2}} \tag{3}$$

$$\omega = 2\pi f \tag{4}$$

and R_p is the oil resistance (ohm).

Statistical Data Analysis

Statistical analysis was performed on the experimental data using SAS version 9.00 (SAS Institute, Inc., Cary, N.C.). Analysis of variance (ANOVA) was conducted to determine if frequency, sludge contamination, and temperature had significant effects on the dielectric constant. Duncan's multiple range test (DMRT) was conducted to compare the means of the treatments. After that, MATLAB version 7.9.0 (The

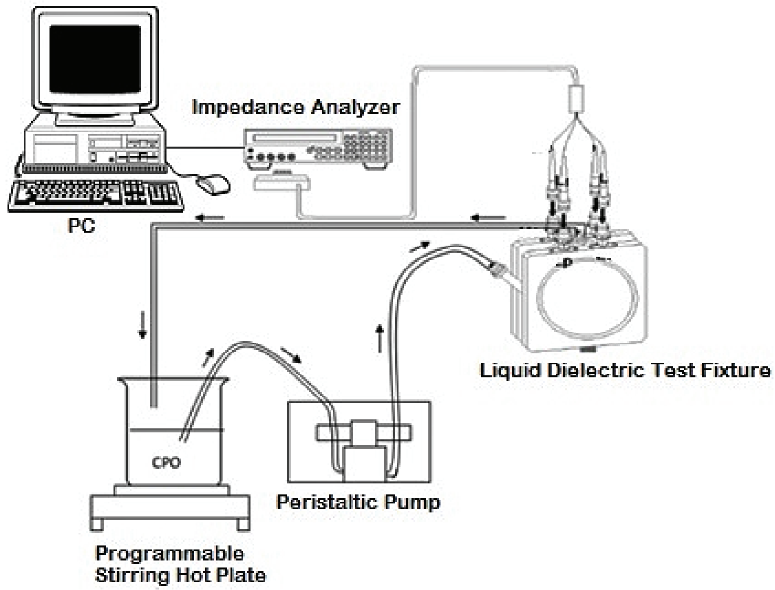

Figure 1. Experimental setup for measuring dielectric properties of CPO.

Mathworks, Inc., Natick, Mass.) was used to perform analysis for CPO contamination prediction. Analysis was performed using principal component regression (PCR) analysis and the multivariate analysis algorithms in PLS Toolbox (Eigenvector Research, Wenatchee, Wash.) for developing a prediction model. The cross-validation was conducted using the random subsets method in which different test sets were determined through random selection of objects in the data set such that no single object was in more than one test set. This procedure was repeated in several iterations. The number of iterations was determined by trial and error. The standard error of cross-validation (SECV) was used as a measure of model performance.

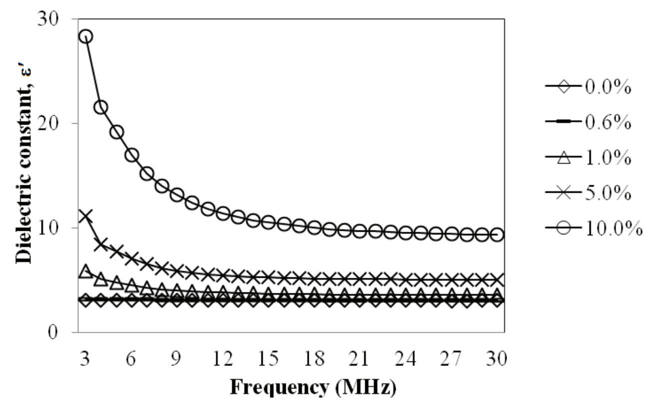

Figure 2. Dielectric constant of CPO at various sludge contamination levels (%) at temperature of 28°C.

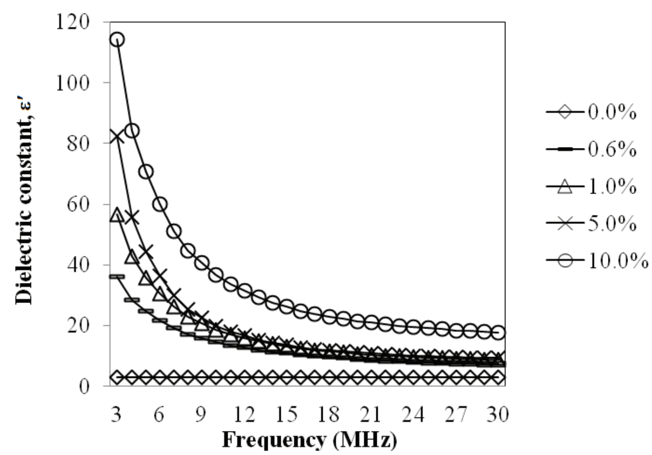

Figure 3. Dielectric constant of CPO at various sludge contamination levels (%) at temperature of 45°C.

Results and Discussion

Dielectric Constant Distributions of CPO

Overall results indicated that there were substantial differences in the dielectric properties of CPO at various temperatures with changes in contamination levels. Results also showed that the dielectric constant of contaminated CPO for all temperature levels had a similar trend of distribution. Representative results for the dielectric constant of various sludge-contaminated CPO samples at 28°C and 45°C are shown in figures 2 and 3. The results clearly indicate that the dielectric constant of contaminated CPO increased with increasing sludge concentration from 0.6% to 10%. This was expected because the sludge had a high moisture content, which increased the polarization effect of the sample and consequently the dielectric constant value.

The dielectric constant of contaminated CPO showed similar frequency dependence for each contamination level. In the frequency range of 3 MHz to around 18 MHz, the dielectric spectra of the contaminated CPO decreased as the frequency increased, and a clear separation in magnitude was observed between the sludge contamination levels. At frequencies beyond 18 MHz, the dielectric constant values were almost constant and reached a minimum value.

Statistical Analysis of Data Using ANOVA

ANOVA showed that frequency, sludge contamination level, and temperature were all highly significant in affecting the means of the dielectric constant ($p < 0.0001$,

Table 1. Overall analysis of variance for the mean dielectric constant of CPO contaminated with sludge.

Source of Variation	Degrees of Freedom	F-Value	p-Value[a]
Frequency	27	44.37	<0.0001
Sludge contamination	4	163.31	<0.0001
Temperature	3	210.11	<0.0001

[a] Significant at 10% significance level or 0.1 probability level. Significant at 5% significance level or 0.05 probability level. Highly significant at 1% significance level or 0.01 probability level.

Table 2. Duncan's multiple range test (DMRT) for the mean dielectric constant of CPO contaminated with sludge at different temperatures.

Treatment	Mean Dielectric Constant, ε′	Duncan Grouping[a]
Measurement of ε′ of pure CPO		
28°C	3.07	a
35°C	3.06	b
45°C	3.04	c
55°C	3.01	d
Measurement of ε′ of 0.6% contaminated CPO		
28°C	3.15	b
35°C	4.22	b
45°C	15.10	a
55°C	13.14	a
Measurement of ε′ of 1% contaminated CPO		
28°C	3.92	b
35°C	5.34	b
45°C	17.23	a
55°C	19.68	a
Measurement of ε′ of 5% contaminated CPO		
28°C	5.80	c
35°C	8.61	c
45°C	19.78	b
55°C	55.35	a
Measurement of ε′ of 10% contaminated CPO		
28°C	12.21	c
35°C	16.78	c
45°C	34.17	b
55°C	63.53	a

[a] Duncan groupings showing different letters indicate that the mean dielectric constants of the treatment are significantly different.

table 1). To further investigate the effects of temperature and sludge contamination, DMRT at each level of sludge contamination was performed to compare the means of the dielectric constant of the CPO at different temperature levels (table 2).

Effect of Temperature on Dielectric Properties of Pure CPO

Based on the results of DMRT, there were significant differences between the mean measured dielectric constants of pure CPO at all temperature levels (table 2). The mean measured dielectric constant of pure CPO decreased from 3.07 to 3.01 as the temperature increased. This result is similar to the results presented by Lizhi et al. (2008), where tested edible oils showed small values of dielectric constant, and the dielectric constant of the oil demonstrated a remarkable decrease with increasing temperature. Temperature had a significant effect on the dielectric properties of pure CPO because temperature affects oil density. Oil density decreases when the temperature increases. A less dense oil contains fewer oil molecules per unit volume. A smaller number per unit volume means that there is less interaction between the oil molecules and the electric field. Therefore, a slight decrease in the value of the dielectric constant was observed.

Effect of Sludge Contamination on Dielectric Properties of CPO

Despite the effects discussed in the preceding section, when sludge contamination was introduced into the CPO, the dielectric constant of the contaminated CPO generally increased as the temperature and contamination level increased. For example, at 18 MHz, the dielectric constant of 55°C CPO increased from around 3.01 to 36.34 with increasing contamination level from 0.0% to 10.0% (fig. 4). As discussed previously, this was expected because the sludge had a high moisture content, which increased the polarization effect of the sample. Temperature increments further energized the polarization effect and consequently the dielectric constant value. Specifically, based on DMRT, the mean dielectric constant of the 5.0% contaminated CPO ranged from 5.80 to 55.35 (table 2), while that of 10.0% contaminated CPO ranged from 34.17 to 63.53 and was found to be significantly different at the 45°C and 55°C temperature levels. On the other hand, in some cases, the mean measured dielectric constants were not significantly different at certain contaminated CPO levels, although the values were quite distinct. For example, at the 10% contamination level, the mean dielectric constants at 28°C and 35°C were 12.21 and 16.78, respectively. Similarly, for 1.0% contaminated CPO, the mean dielectric constants were 17.23 and 19.78 at 45°C and 55°C, respectively.

The effect of sludge contamination was also observed from DMRT (table 3). Generally, the dielectric constant of sludge-contaminated CPO increased from around 3.17 to up to 63.53 as the contamination level increased at different temperature levels. Overall, based on DMRT, there were significant differences between the dielectric constants of pure CPO and contaminated CPO at different contamination levels. In a few cases, the dielectric constants were not significantly different as the contamination level increased. This effect was observed, for example, at 28°C for the 0% to 0.6% contamination levels, at 45°C for the 0.6% to 5% levels, and at 55°C for the 0.6% to 1% levels.

Sludge Contamination Prediction Using PCR and PLS Analysis

PCR analysis was used to develop models for sludge contamination classification at different temperature levels. Overall, the results showed that dielectric measurements are sensitive to CPO temperature, and when temperature was treated as a known parameter, the PLS and PCR models showed substantially good potential to predict contaminant levels.

The PCR sludge contamination classification models gave coefficient of determination (r^2) values in the range of 0.91 to 0.98 for individual temperatures. CPO with sludge contamination yielded the most accurate result at 28°C when relating predicted sludge contamination to measured sludge contamination, with an r^2 of 0.98 (fig. 5). The prediction model for CPO with sludge contamination at 28°C yielded the most accurate result because there was more interaction between the oil molecules and the electric field at this temperature compared to higher temperatures. Therefore, when contamination exists in oil at this temperature, it is more detectable compared to higher temperatures.

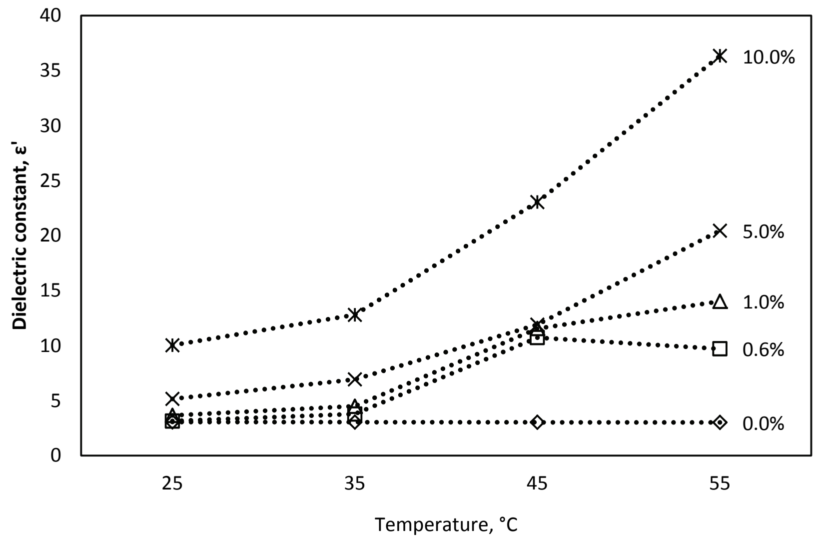

Figure 4. Dielectric constant of CPO for different contamination level (%) at 18 MHz.

Table 3. DMRT on the mean dielectric constant of CPO at different sludge contamination levels.

Treatment	Mean Dielectric Constant, ε′	Duncan Grouping[a]
Measurement of ε′ at 28°C		
Pure CPO	3.07	d
0.60%	3.15	d
1.00%	3.92	c
5.00%	5.80	b
10.00%	12.21	a
Measurement of ε′ at 35°C		
Pure CPO	3.06	e
0.60%	4.22	d
1.00%	5.34	c
5.00%	8.61	b
10.00%	16.78	a
Measurement of ε′ at 45°C		
Pure CPO	3.04	c
0.60%	15.10	b
1.00%	17.23	b
5.00%	19.78	b
10.00%	34.17	a
Measurement of ε′ at 55°C		
Pure CPO	3.01	d
0.60%	13.14	c
1.00%	19.68	c
5.00%	55.35	b
10.00%	63.53	a

[a] Duncan groupings showing different letters indicate that the mean dielectric constants of the treatment are significantly different.

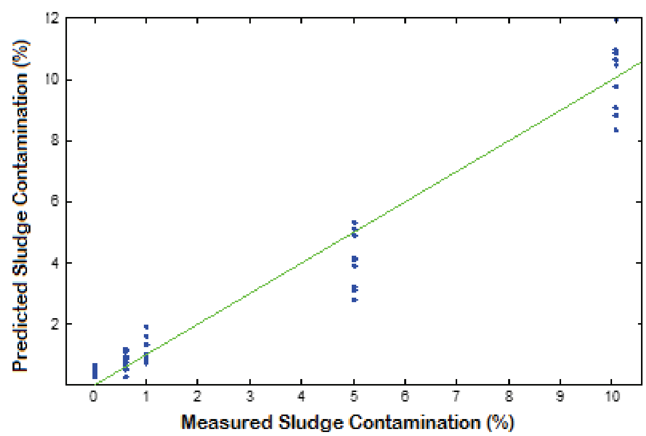

Figure 5. Predicted versus measured sludge contamination of CPO at 28°C using common PCR regression model (r^2 = 0.98).

Table 4. Cross-validation performance of PLS calibration model for sludge contamination prediction in CPO at different temperatures.

Temperature (°C)	PLS Calibration Model No. of Latent Variables	PLS Calibration Model SECV (%)	PCR Analysis r^2
28	4	1.04	0.98
35	3	1.10	0.96
45	4	1.32	0.96
55	1	1.61	0.91

PLS analysis was used to develop models that related dielectric constant spectra to sludge contamination and showed substantial results in cross-validation performance as measured by SECV for all temperature levels of CPO (table 4). The PLS models developed by correlating dielectric constant spectra to sludge contamination gave SECV values ranging from 1.04% to 1.61%. Overall, the best result was obtained at 28°C with the lowest value of SECV of 1.04%. This showed that, based on our experimental data, sensing prediction can be estimated up to about 1% accuracy.

Conclusions

In this study, the ranges of CPO dielectric constants could be easily distinguished at different contamination and temperature levels. The dielectric constants of pure and contaminated CPO both exhibited a general pattern that had a maximum value at lower frequencies and then decreased significantly as the frequency increased.

Generally, there was no significant difference in the mean measured dielectric constants of CPO at 28°C and 35°C for all contamination levels studied. However, there were generally significant differences in the mean measured dielectric constants as the temperature increased to

55°C ($p < 0.0001$). When sludge was introduced into the CPO with increasing contamination levels (0% to 10%), the dielectric constants at frequencies ranging from 3 to 30 MHz increased from 3.01 to 63.53. Generally, there were significant differences between the dielectric constants of pure and contaminated CPO ($p < 0.0001$).

The results showed that dielectric measurements are sensitive to CPO temperature, and when temperature was treated as a known parameter, the PLS and PCR models showed strong potential to predict contaminant levels. Classification of sludge contamination yielded very substantial correlations, with r^2 values ranging from 0.91 to 0.98 and SECV values ranging from 1.04% to 1.61%. The best result was obtained at 28°C with the lowest SECV of 1.04%.

The results from this study indicate that dielectric spectral measurements were useful and have strong potential for use as inputs in the development of a sensing system for estimating CPO sludge contamination. This study was conducted at a generally low frequency range (3 to 30 MHz); therefore, the instrumentation required for such measurements is relatively inexpensive for industrial application. The application of this method to the palm oil industry is relatively new, which confers a prospective role upon this study, its general aim being to assess the viability of using dielectric spectroscopy for *in situ* monitoring of CPO sludge contamination.

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