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Direct Analysis of Infant Formula Via Laser Induced Breakdown Spectroscopy

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Abstract: For infant consumers' healthy and strong growth, the quantity of nutrition in the infant formula must be controlled. Therefore, the work presented here aims to analyse an elemental composition in infant formula. In order to analyse the nutritional value in infant formula, we proposed employing the laser induced breakdown spectroscopy (LIBS) approach combined with principal component analysis (PCA) as their analysing tool to directly study elemental composition in infant formula. PCA results shows the first principal component accounts for around 83.3 percent of total variation, while the second principal component accounts for nearly 15.2 percent. The PC1 is defined by Calcium and Sodium elements, whereas the PC2 is characterised by Magnesium and Potassium elements, according to NIST database mapping and PCA analysis. The components appear to play a major role in the nutritional value of infant formula. We can clearly state that the LIBS technique has the capacity to conduct direct analysis of food sample due to its capability of analysing elemental composition in infant formula.

Keywords: Infant Formula, Laser-Induced Breakdown Spectroscopy, LIBS, PCA

1. Introduction

Infancy is a stage that is crucial for the infant's growth and development, which requires various nutrition to boost their health condition. However, breastfeeding needs a lot of commitment from the mother because babies usually often need to be fed in a day in an early stage. The presence of infant formula that acts as a complete or partial substitution for human milk, primarily for food used in infants will reduce and resolve problems that occur among parents. There are many other components in infant formula that important for the growth of the babies such as calcium (Ca), magnesium (Mg), sodium (Na) and potassium (K). During the first four months of development, human milk had a 60 percent

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absorption efficiency and baby formula had a 40 percent absorption efficiency [1]. Calcium is the most plentiful in the body because they are responsible for the different cell activities of the bones, which cause bone strength and rigidity [2]. Magnesium is essential for children's growth, development, and energy production [3]. Sodium is an electrolyte that controls fluid balance and aids in nerve and muscle function modulation [4]. Potassium also should be included in a baby's diet because potassium aids in regulating of fluid balance in the body and is also essential for keeping a healthy blood pressure. It is vital to understand the components of infant formula. This is because, in many nations, poor nutrition can contribute to a growing public health concern. Poor nutrition raises the risk of diseases and is responsible for one-third of the estimated 9.5 million deaths in children under the age of five that occurred in 2006 [5]. Stunting is caused by malnutrition in the first two years of life, resulting in an adult who is several centimeters shorter than his or her potential height [6]. Adults who were malnourished as children have been shown to have poor intellectual performance [7].

Laser-induced breakdown spectroscopy or LIBS is the technique for analysing and characterisation of materials. There are several reasons on why LIBS technique is used for infant formula analysis. The method is straightforward to use and does not necessarily involve sample preparation [8]. LIBS technique can be one of the most rapid analytical technique for analysing and identifying the compositions of elements in the target sample [9]. LIBS, as the general technique, can test many elements at once and can even detect some of the lighter elements that other technique can't detect. This is necessary for the analysis since it speeds up the process, and since the sample will frequently contain other elements, being able to identify these elements will make it easier to distinguish between various samples.

LIBS is the atomic emission spectroscopic technique that generates plasma from a material using a concentrated pulsed laser beam [10]. With high-energy laser pulses converging to LIBS samples, the high-energy material density at the laser position will make the material high in temperature and high in electrons. The plasma is formed by laser light on the sample surface from the laser system. The image capture card collects and transmits a plasma image to the image processing device after collecting the plasma by the high-speed cameras attached to the synchroniser. Changes in the laser's energy intensity may be reflected in the plasma area as the laser's energy changes the spectral line, allowing the laser system's stability to be monitored [11]. In LIBS, laser source signal fluctuation can decrease sensitivity and accuracy [12]. Although LIBS has lower detection limits (LODs) and precision compared to ICP-OES, it is extremely sensitive in absolute terms [13]. Flame atomic absorption spectrometry (FAAS) and digital image-based flame atomic emission spectrometry (DIB-FES AES) also can be used in infant formula analysis [14]. LIBS, on the other hand, is preferred since these analytical technique are destructive and require the sample to be in the flame to produce radiation.

In this study, the elemental composition in infant formula which is Mg, Ca, K, and Na was analysed from various infant formula. A light (plasma) that produced as the laser beam hits on the surface of infant formula was recorded, and their light intensity was considered to determine the type of elemental composition (Mg, Ca, K, and Na) in each of the samples.

2. Materials and Methods

Three samples (A, B, and C) of infant formula from different brands are used in this study collected in Johor, Malaysia. To ensure that the samples are of diverse quality, each of the three used a different brand of infant formula, ranging from their popularity and prices from expensive to the cheapest brand of infant formula.

Figure 1: Nutritional Information of Infant Formula.

Figure 1 shows the nutritional information of infant formula sample that used in this study. These are the key components that make up infant formula and help to support the growth of the babies that consume it. There are various elements in infant formula, such as fat, linoleic acids, protein, and fat-soluble A, D, E, K, and B vitamins, but only calcium, potassium, sodium, and magnesium were evaluated.

Table 1: Sample Preparation.

Sample	Before Preparation	After Preparation
A		
B		
C		

Based on the Table 1, each of the sample of the milk was prepared in palette form. A small amount of infant formula powder (3 - 5g) was placed in hydraulic pressing to transform it into the palette form. Each sample is placed on its petri dish. The analysis of palette samples involves the minimum amount of sample preparation, which saves time throughout the experiment. The schematic diagram for this LIBS system is shown in Figure 2. A Q-switched Nd: YAG laser was used in this study as a source of energy.

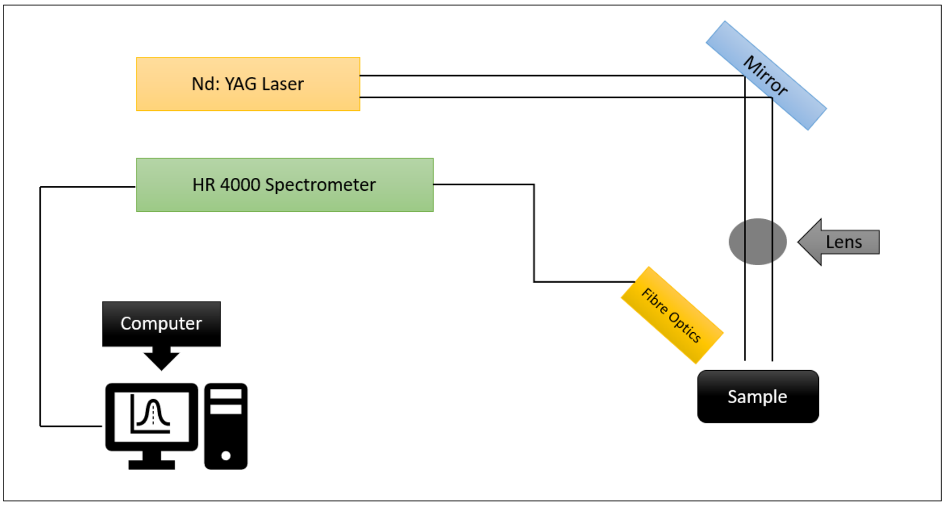

Figure 2: Experimental Setup for LIBS Analysis.

A Q-switched Nd: YAG laser with a wavelength of 1064nm was focused onto the powdered infant formula through a convex lens with focal length of 9cm along with the laser energy of 200 mJ. The laser beam was targeted into the sample and forming the plasma. For each sample, ten measurements were performed to get the average spectrum. The plasma spectrum formed from the experimental procedure was captured using an HR 4000 spectrometer. Then, the data were analysed by using the Principal Component Analysis (PCA) method.

3. Results and Discussion

Figure 3 illustrates the emission spectra of infant formula used in this study.

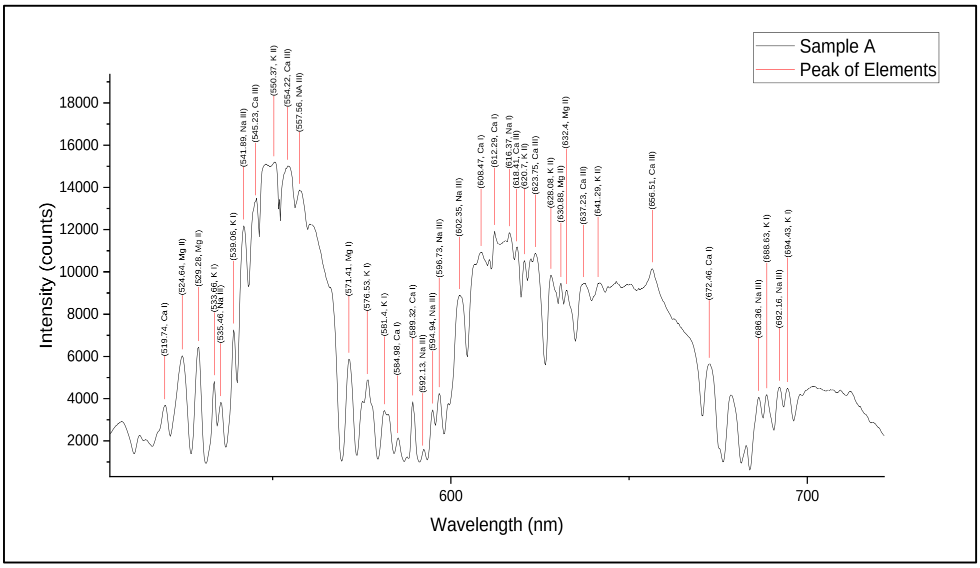

Figure 3: LIBS Spectrum of Sample A of Infant Formula Obtained by Optical Emission Spectroscopy from NIST database.

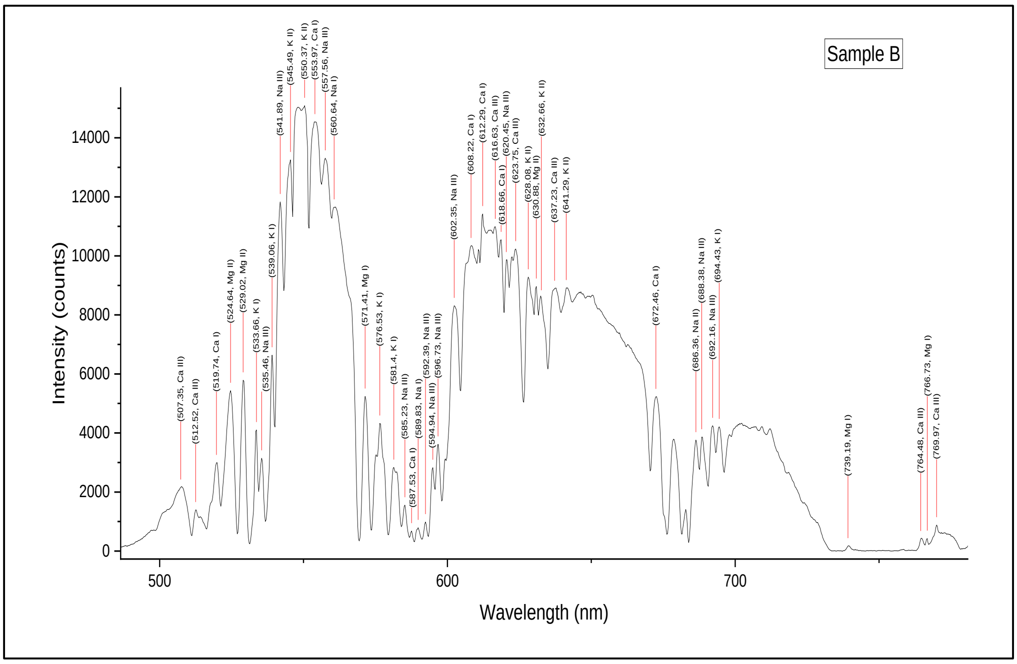

Figure 4: LIBS Spectrum of Sample B of Infant Formula Obtained by Optical Emission Spectroscopy from NIST database.

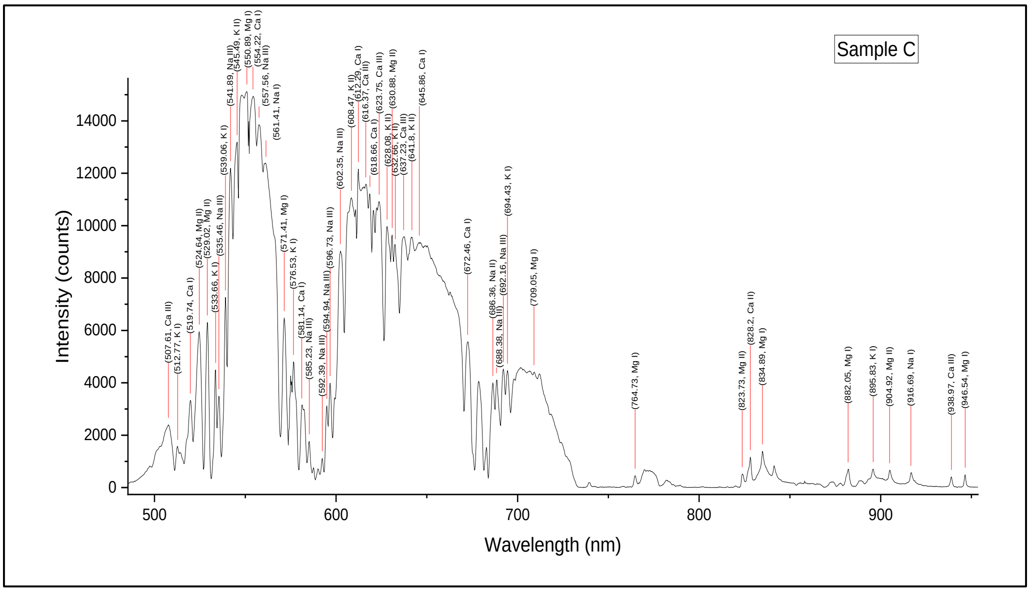

Figure 5: LIBS Spectrum of Sample C of Infant Formula Obtained by Optical Emission Spectroscopy from NIST database.

Figure 3, Figure 4 and Figure 5 show the graphs for all the three samples' LIBS spectrum. For all three samples, sample A, B and C infant formula, the highest peak of the spectrum is at the range of 500nm to 600nm wavelength.

Table 2: List of Identified Emission Line and its Elements.

Item	Elements	Wavelength (nm)	Reference
1	Calcium, Ca	I-519.74, I-553.97, I-581.14, II-828.20, III-507.35, III-507.61	[15] [16]
2	Sodium, Na	I-560.64, I-561.41, I-589.83, III-535.46, III-541.89, III-557.56	[16]
3	Potassium, K	I-512.77, I-533.66, I-539.06, II-545.49, II-550.37, II-608.47,	[17] [16]
4	Magnesium, Mg	I-550.89, I-571.41, I-709.05, II-524.62, II-529.02, II-529.28,	[15] [16]

Table 2 shows the list of identified elements according to their emission line. Each of the wavelength obtained from the LIBS spectrum refers to its own elements. The elements that were detected from this study was Calcium (Ca I, Ca II, Ca III), Sodium (Na I, Na III), Potassium (K I, K II), and Magnesium (Mg I, Mg II). All these elements were obtained based on the NIST database. The elements that were found correspond to the nutritional information provided by the manufacturer.

The light spectrum that is produced contains a lot of data, so using PCA it can simplify to characterise the Mg, Ca, K, and Na and other components. The observations in the resulting vectors are uncorrelated orthogonal basis sets with a linear combination of variables. The relative scaling of the influences the original variables PCA. PCA is a dimensionality-reduction approach that decreases the dimensionality of big data sets by converting a large collection of variables into a smaller one that retains the majority of the data in the large set. Because PCA is the simplest of the real eigenvector-based multivariate approaches, it is chosen to investigate the elemental composition in infant formula.

The strength of each characteristic's influence on a major component is shown in a loading plot. The loading plot is used to discover out which variables have the most influence on each component. Loadings might be anything between -1 and 1. Loadings near -1 or 1 imply that the variable has a significant impact on the component. Loadings that are close to zero imply that the variable has a minor impact on the component. Figure 6 shows the loading plot for the infant formula samples in this research.

According to the principal component analysis results, the first principal component accounts for around 83.3 percent of the total variation, while the second principal component accounts for nearly 15.2 percent. According to NIST database mapping and PCA analysis, the PC1 is defined by Calcium and Sodium elements, whereas the PC2 is characterised by Magnesium and Potassium elements. This is related to the opposing effect of Ca and Na content with strong negative loadings, whereas Mg and K elements with high positive loadings made major contributions in PC2. These variables were correlated and also contributed to the variances in these PCs. As a result, the components appear to play a major role in the nutritional value of infant formula.

A graphical representation known as a scree plot is a standard way for calculating the number of PCs to be maintained. A Scree Plot is a basic line segment plot that displays the eigenvalues of each PC. Figure 7 shows the scree plot of PCA in infant formula samples.

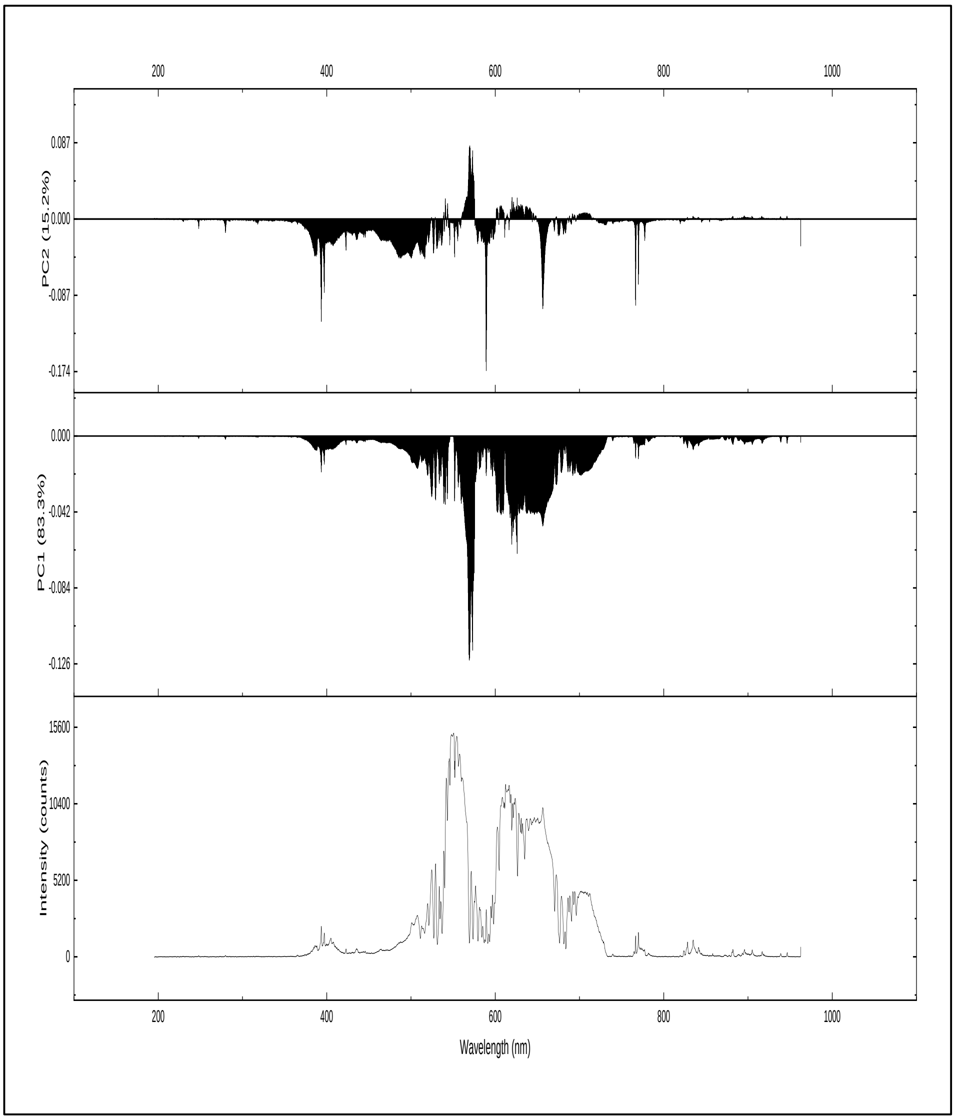

Figure 6: Loading Plots for Principal Component of Infant Formula Samples.

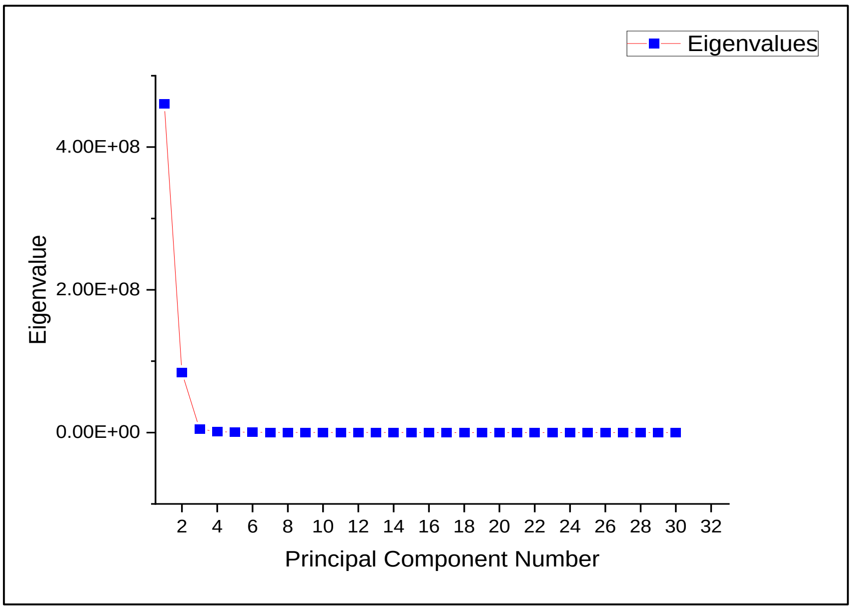

Figure 7: Scree Plot for Principal Component in Infant Formula Samples.

Based on Figure 7, the eigenvalues are plotted on the y-axis, while the number of factors is plotted on the x-axis. It always has a downhill curvature. Most scree plots have a similar form, starting high on the left, quickly descending, and then levelling out at some point [18]. It is because the first component typically explains a large portion of the variability, the following few components a moderate proportion, and the final components only explain a small portion of the overall variability. The scree plot criterion searches for the curve's "elbow" and chooses all components right as the line begins to flatten out.

The principal components or partial least squares factors are plotted in a score plot. This demonstrates how each sample is projected onto the principal component. Figure 8 shows the score plot of infant formula referred to their colour.

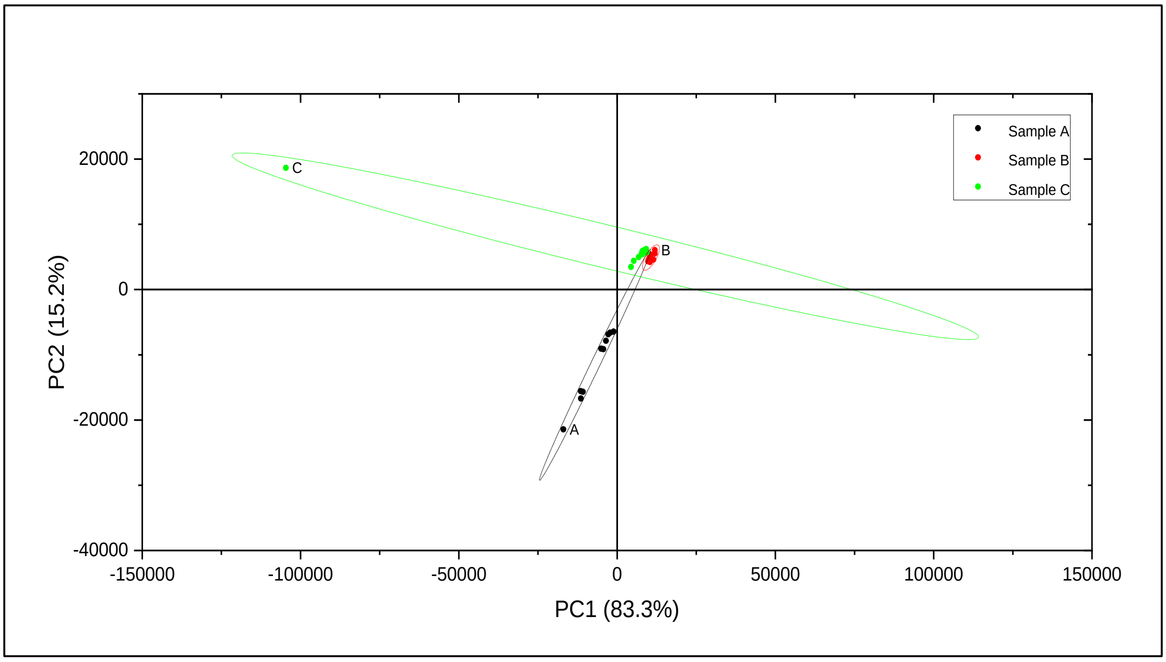

Figure 8: Score Plot for Principal Component in Infant Formula Samples.

The placement of infant formula brands in the multivariate space of three principal component score vectors is illustrated in Figure 8's score plot. There are three distinct groups of scores according to the samples. The first two PCs of a data set about infant formula elemental profiles are plotted in a PCA score plot. It describes a visual representation of how the elements in those samples are related to one another. The first component accounts for 83.3% of the variance, whereas the second only accounts for 15.2%. The darkest one is the sample A which mainly in PC1. The green one, sample C and the red one, which is sample B, mainly has the highest score on PC1. However, one from the sample C has the least variance with the PC2.

Based on the overall findings of several spectrum analyses, PCA proven to be a viable approach for selecting the most beneficial factors and promptly pointing out the link between the variables. Furthermore, PCA enables quick differentiation, categorisation, and identification of the associated LIBS spectrum components. The relationship between spectrum features and variables identifies the existence of elements, and hence the infant formula groupings. In addition, the scores plot also shows large gap between groups.

4. Conclusion

The elements that are calcium, potassium, sodium, and magnesium present in the different infant formula brands are successfully detected using the laser-induced breakdown spectroscopy (LIBS) technique. For all the three samples, sample A, B and C infant formula, the highest peak of the spectrum is at the range of 500nm to 600nm wavelength. According to the elements found, each of these element's correlates to the nutritional information provided by the manufacturer.

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