

Spline Regression on Percentage of Reflectance Based on Wavelength of Lithium Niobate (LiNbO₃) Doped with Ruthenium Oxide (RuO₂)

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Abstract

The characteristics of LiNbO₃ are interesting to learn deeply with statistical analysis to identify unique things to optimize its potential in daily lives. The reflectance of LiNbO₃ is influenced by the wavelength which can be described as a regression model in y = f(x) with x is wavelength and y is the percentage of reflectance. Using the parameters of Spline regression, the wavelength can be cut into several segments and each segmentation would have its regression model. This study aimed to estimate percentage of reflectance and to learn the wavelength segmentation at various concentrations (0, 2, 4, and 6 %) of LiNbO₃ doped with RuO₂. The results show that the influence of the doping process exists and optimum knots is four knots in the second order. Thus, there are five segmentation and regression models for each concentration. Based on minimum/maximum local for each segment, the lowest minimum of reflectance in the first and fifth segment are produced by LiNbO₃. In the second and third segments, LiNbO₃ doped with RuO₂ 6% has the highest maximum, while in the fourth segment, the lowest minimum was obtained at LiNbO₃ doped with RuO₂ 6%. Based on its concentration, the lowest local minimum among all segments of LiNbO₃ is 25.06 percent and the highest is 32.66 percent. For concentrations of 6%, the lowest minimum is 36.90 percent and the highest maximum is reached at 72.305 percent.

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1. Introduction

Lithium Niobate (LiNbO₃) consists of compounds lithium, niobium, and oxygen. LiNbO₃ is a synthetic or manmade material. By Chemical Solutions Deposition (CSD) method, thin films of LiNbO₃ are made by placing the solution on the substrate to be rotated at a certain speed by means of spin coating [1]. The characteristics of LiNbO₃ are closely related to its constituent substances, its mineral structure, and the molecular shape of this element itself. It is an important ferroelectric because it has excellent piezoelectric, electro optical, pyro electrical and photorefractive [2]. In the past few years, $LiNbO_3$ thin films have been widely applied in various fields. LiNbO₃ acts as the main ingredient in optical waves (electro-optics, photo refraction, and non-linear optics) for hologram recording and second harmonic generation (SHG), the telecommunication industries such as cellphones, piezoelectric sensors, solar cell materials, various acoustic applications, and much more. LiNbO₃ is also a constituent substrate in modular optics to convert electrical signals to optical signals [3]. Therefore, further research on LiNbO₃ is very important to understand its characteristics deeply. So far, more research on the characteristics of LiNbO₃ has been carried out in the laboratory, even though the research data can be further processed by statistical analysis. The characteristics are interesting to learn with statistical analysis to identify unique things that can be used to optimize the potential of their use in daily lives. Further studies are not limited to LiNbO3 but rather expanded from LiNbO3 doped with various concentrations of thin films of Ruthenium Oxide (RuO_2). This mixing process in the experiment design was carried out by [4], was done as a form of treatment and looking at the performance of LiNbO3. LiNbO3 as a ferroelectric can reflect itself against the received light (reflectance). Reflectivity shows the amount of electromagnetic wave energy reflected by objects [5], can be measured by a UV-VIS spectrophotometer that utilizes near ultraviolet light and visible light. Reflectance is influenced by the wavelength which can be described in the function y = f(x) with x being the wavelength, while y is the percentage of reflectance of LiNbO₃. The relationship between wavelength and percentage reflectance of LiNbO₃ can be described in a regression equation. However, the response produced is not always linear, so the non-parametric regression approach is more appropriate. The coefficients are also not unique to some segments due to differences in data patterns. In addition, the estimation of this type of data pattern cannot be analyzed by parametric models because in the parametric models there are assumptions that must be fulfilled. This case can be approached with Spline Regression, which can handle the pattern of relationships between two variables in a good visual curve, have high flexibility, and are able to adjust to the characteristics of the data [6]. Previous studies related to this case, studied the characteristics of $LiNbO_3$ with ARIMA, stating that when $LiNbO_3$ doped with La_2O_3 , it could increase the percentage of absorption [7]. According to other studies, the best model for estimating SrTiO₃ is a spline truncated regression of the first order with 42 knots and a for SrTiO₃ doped with RuO₂ is spline truncated regression of the first order with 37 knots [8]. From other research, Ba2TiO3's wavelength segment was divided into three, then performs a regression analysis according to the data pattern (linear or quadratic). The model is quite good with a large Rsquare, but the segments are made manually, making it difficult when the data used is fluctuating [9]. Other studies suggest that LiNbO₃ doped with variations concentrations of the RuO_2 (0, 2, 4, and 6%) has an effect on refractive index and energy gap data, potentially becoming the forerunner of the light sensor material [4].

Spline regression is a non-parametric analysis with the concept of polynomials that have segmented properties [10]. Using these parameters, the wavelength can be cut into several segments and the best regression based

regression models can be formed for each wavelength segmentation. The segmented analysis is necessary because, in its application of LiNbO₃, each tool requires a different range of wavelength. That is why a segmented model is supposed to give a more accurate estimation. Wavelength segmentation is formed from the cut point. The cut point is the value of x (in the explanatory variable) where the slope forms a linear and non-linear pattern [9]. It can also be said as the point where changes in data patterns occur, for example from concave to convex. Deeper exploration in this way is expected to be able to present more recommendations related to the use of LiNbO₃ in each wavelength segment. Based on the description above, this study aimed to estimate percentage of reflectance and to learn the wavelength segmentation at various concentrations (0, 2, 4, and 6 %) of LiNbO₃ doped with RuO₂.

2. Data and Method

LiNbO₃ data in this study is secondary data from the Department of Physics of the Bogor Agricultural Institute in 2017. LiNbO₃ is given four variations of Ruthenium Dioxide (RuO₂), namely 0, 2, 4, and 6%. Data was taken with a UV-VIS spectrophotometer. The UV-VIS spectrophotometer measures the relative amount of reflected light (reflectance) by the LiNbO₃ thin film. The optical characteristics of the thin film will provide information about the percentage of reflectance at different wavelengths. The reflectance percentage is the response variable, while the wavelength as an explanatory variable is in the range of 450.3 to 900.9 nm. Then, the data was analyzed by Spline Regression. Spline regression is a non-parametric regression consisting of pieces of polynomials with a particular order and connected to tie points or cut points. The abscissa value of the tie point is commonly called a knot. Knots were interpreted as a focal point in spline functions, so that segmented curves at that point were formed [11]. An optimum knot will be chosen from various knots that have been tried. This optimum knot is used as a parameter to estimate.

Spline functions of p-th order [12] can be expressed as follows

$$f(x_i) = \beta_i x_i^0 + \beta_1 x_i^1 + \beta_2 x_i^2 + \dots + \beta_p x_i^p + \sum_{k=1}^K \beta_{p+k} (x_i - K_k)_+^p$$
(1)

with

 β = regression coefficients,

x = explanatory variable,

k = number of knot,

and

$$(x_{i} - K_{k})_{+}^{p} = \begin{cases} (x_{i} - K_{k})^{p}, x \ge K_{k} \\ 0, x < K_{k} \end{cases}$$
(2)

for $a < K_1 < K_2 < \cdots < K_k < b$ with a and b each of them is the smallest and largest value of the data. The "+" indicates that only $(x_i - K_k)^p \ge 0$ will be taken, while the value <0 is considered to be 0. If the number of

observations is "n", the function matrix is shown below

$$\begin{bmatrix} f(x_1) \\ f(x_i) \\ \vdots \\ f(x_n) \end{bmatrix} = \begin{bmatrix} 1 & x_1^{1} & x_1^{2} & \dots & x_1^{p} & (x_1 - K_1)_{+}^{p} & \dots & (x_1 - K_k)_{+}^{p} \\ 1 & x_2^{1} & x_2^{1} & \dots & x_2^{p} & (x_2 - K_2)_{+}^{p} & \dots & (x_2 - K_k)_{+}^{p} \\ \vdots & \vdots & \vdots & \vdots \\ 1 & x_n^{1} & x_n^{2} & \dots & x_n^{p} & (x_n - K_n)_{+}^{p} & (x_n - K_k)_{+}^{p} \end{bmatrix} \begin{bmatrix} \beta_0 \\ \beta_1 \\ \vdots \\ \beta_p \\ \beta_{(p+1)} \\ \beta_{(p+2)} \\ \vdots \\ \beta_{(p+K)} \end{bmatrix}$$
(3)

or can be express as

$$\hat{f}(X_i) = \mathbf{X}\boldsymbol{\beta} \tag{4}$$

In spline regression between segments separated by knots. Therefore, the location of knots, and the number of knots will determine the goodness of spline regression in the data. The optimum knots are selected based on the smallest *Generalized Cross Validation* (GCV) [13].

$$GCV = \frac{MSE}{(n^{-1}tr[\mathbf{I} - \mathbf{H}])^2}$$
(5)

where

$$MSE = \frac{1}{n} \sum_{i=1}^{n} (Y_i - \widehat{Y}_i))^2$$
(6)

and

$$\mathbf{H} = \mathbf{X}_{\mathbf{K}} (\mathbf{X}_{\mathbf{K}}^{\mathrm{T}} \mathbf{X}_{\mathbf{K}})^{-1} \mathbf{X}_{\mathbf{K}}^{\mathrm{T}}$$
(7)

with

 $\mathbf{X}_{\mathbf{K}}$ = the explanatory variable matrix in spline regression with knots k

The methods consist of following step

- 1. Data exploration
- 2. Determine the number and location of knots with the help of the freeknotsplines package [14] on the R program
- 3. Building spline regression in first order (linear) and second order (quadratic) with the syntax of the R program [15].
- 4. Choose the best spline regression based on GCV.
- 5. Form the most significant wavelength segment based on the best spline regression
- 6. Exploration of the wavelength segmentation formed at various LiNbO₃ doped with RuO₂

3. Results and Discussions

3.1 Data Exploration

The percentage reflectance of LiNbO₃ is influenced by the wavelength that is fired. Data plot between reflectance percentage as y-axis or response variable and wavelength as x-axis or explanatory variable at various concentrations (0, 2, 4, and 6%) LiNbO₃ doped with RuO_2 is shown in Figure 1. It can be seen exploratively in Figure 1 that the fluctuation of percentage reflectance is not too large but has a different pattern between concentrations. The percentage of reflectance at concentrations of 0 and 2% generally has the same pattern, forming a pattern with a rising trend, then going down. The percentage of reflectance tends to experience an upward trend for each concentration at a wavelength of nearly 500 nm. Data has a downtrend when the wavelength enters the 700 nm number. At a concentration of 4%, at the beginning of the data appears to have formed a downward pattern, then shows an upward trend. At a concentration of 6% tend to form a rising trend since the initial data and had established a pattern decreased when wavelength around 700 nm, but rising, again.



Figure 1: LiNbO3 doped with RuO2 (a) 0%, (b) 2%, (c) 4%, (d) 6%

Figure 1 shows the differences in percentage produced and the range of reflectance percentages between concentrations. That is, there is an influence given by the doping process between LiNbO₃ and RuO₂. Based on table 1, after the doping process, it appears that the percentage of reflectance increases compared to before (0%). The highest increase occurred at a concentration of 6%, while the lowest was a concentration of 4%. Based on the first quartile and the minimum value, it appears that the concentration of 4% has a reflectance percentage lower than the concentration of 2%, but continues to rise every time the wavelength increases, seen from the median, mean, quartile 3, and the maximum value of concentration of 4% higher than at a concentration of 2%. This distinction reinforces that doping process has a significant influence in the form of a percentage difference in reflectance produced. The highest reflectance percentage (maximum) at 0, 2, 4 and 6%

concentrations of 34.05, 45.91, 56.00 and 78.95 (in percent), respectively.

Statistic	Percentage of Reflectance				Wavelength	
	0%	2%	4%	6%		
Minimum	25.20	36.27	25.31	41.58	450.3	
Quartile 1	31.58	40.42	36.29	60.03	570.0	
Median	32.24	42.40	45.13	70.87	685.0	
Mean	31.76	42.27	42.85	65.98	681.9	
Quartile 3	33.45	44.54	49.05	73.13	795.3	
Maximum	34.05	45.91	56.00	78.95	900.9	

Table 1: Summary of Percentage of Reflectance and Wavelength

3.2 Spline Regression

Selection of the number of knots is done by looking at the pattern in the data, for reasons of simplicity of the model can be used the number of knots slightly. Figure 2 shows the movement of GCV changes to the location and number of knots up to ten with the freeknotsplines package in the R program.



Figure 2: Movement of GCV LiNbO₃ Doped With RuO₂ (a) 0%, (b) 2%, (c) 4%, (d) 6%

According to Figure 2, it appears that GCV changes occur as the number of knots increases for each order. The most significant changes in GCV occurs up to four knots in all orders. Figure 2 also shows that, in many knots greater than four, the changes that occur are relatively small in all orders. Therefore, the optimum knots that will be used in the analysis are four knots. The point of the knot is a cutoff point that forms a piece of

wavelength polynomial. The pieces formed from the results of the spline smoothing with four point knots will be regressed and form a spline regression model. The model chosen is a model with four knots in the order that gives minimum or lowest GCV.

Concentration	1 st Order	2 nd Order
0%	0.034	0.014
2%	0.028	0.020
4%	0.037	0.024
6%	0.082	0.026

Table 2: GCV for First and Second Order with Number of Knot=4

Table 2 shows the minimum GCV at four knots produced by second order (squared) for all concentrations with GCV for 0, 2, 4 and 6% concentrations of 0.014, 0020, 0.024 and 0.024 respectively. Based on these results, the best regression model is given by second order. The location of the knots according to the best order can be seen in table 3.

 Table 3: Location of Knot Points in Second Order

Concentration						
0%	2%	4%	6%			
502.94	496.71	493.43	534.77			
581.97	646.08	729.50	643.93			
585.14	790.35	742.57	778.59			
751.60	855.44	860.21	849.65			

Wavelength segments from spline quadratic regression (2^{nd} order) as the best regression are formed based on knot points as cut points, so in this study there are five wavelength segments, as presented in table 4.

Table 4: Wavelength Segment

Segment	Concentration						
	0%	2%	4%	6%			
1	< 502.94	< 496.71	< 493.43	< 534.77			
2	502.94 - 581.97	496.71- 646.08	493.43 - 729.50	534.77 - 643.93			
3	581.97 - 585.14	646.08 - 790.35	729.50 - 742.57	643.93 - 778.59			
4	585.14 - 751.60	790.35 - 855.44	742.57 - 860.21	778.59 - 849.65			
5	≥ 751.60	≥ 855.44	≥ 860.21	≥ 849.65			

Based on the table above, it appears that the position of knots between concentrations varies. The position of the first knot is in the range of 400-500s or is located in the first quartile of wavelengths. Besides, the position of

the fourth knot is in the third quartile, except the concentration of 0% which is in the second quartile. At concentrations 0 and 4% correspond to two knots close together. The knots are the second and third knots, which are 581.97 and 585.14 for concentrations of 0% and 729.50 and 742.57 for concentrations of 4%. The position of the adjacent knots occurs because the data fluctuations around the range are greater, so the data patterns in that range often change according to the distribution of data.

Table 5: shows that predictive values are not much different from the original data. The MSE produced is also

 relatively small, so it can be said that the model produced is quite good.

Statistic	0%		2%		4%		6%	
	A	D. 1. 1. 1	A	D. 1. 1. 1	A . (. 1	Des 1 ¹ et a 1	A . (. 1	D. 1. 1. 1
	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted
Min	25.20	25.68	36.27	36.48	25.31	25.77	41.58	32.39
Q 1	31.58	31.62	40.42	40.18	36.29	33.94	60.03	53.67
Median	32.24	32.19	42.40	42.07	45.13	43.91	70.87	69.71
Mean	31.76	31.76	42.27	42.03	42.85	41.60	65.98	63.47
Q 3	33.45	33.35	44.54	44.54	49.05	48.73	73.13	73.15
Max	34.05	34.01	45.91	46.55	56.00	56.19	78.95	78.98
Std.	2.209	2.206	2.530	2.526	8.452	8.451	10.296	10.295
Dev.								
X when	458.32		451.05		454.33		450.32	
Y min								
X when	652.05		630.88		900.86		900.86	
Y Max								
MSE	0.014		0.019		0.023		0.025	

1. LiNbO₃ doped with RuO₂ Concentration 0%

At this concentration, spline regression produces adjusted R-Square of 0.9972, meaning that 99.72% of the percentage reflectance can be explained by the model. All segment has significant intercept, coefficient of x, and coefficient of x^2 .

 $\hat{Y} = \begin{cases} 214.3 - 0.8345x + 0.00092x^2, & x < 502.94 \\ -147.417 + 0.603908x - 0.00051x^2, & 502.94 \le x < 581.97 \\ 1231.05 - 4.133x + 0.00356x^2, & 581.97 \le x < 585.14 \\ -59.7579 + 0.278628x - 0.00021x^2, & 585.14 \le x < 751.60 \\ 183.15 - 0.367748x - 0.00022x^2, & x \ge 751.60 \end{cases}$

2. LiNbO₃ doped with RuO₂ Concentration 2%

This concentration has *adjusted R-Square* = 0.997, and from t-test, it already has significant intercept, coefficient of x, and coefficient of x^2 .

$$\hat{Y} = \begin{cases} 338.7 - 1.318x + 0.0014x^2, & x < 496.71 \\ -130.07 + 0.5695 - 0.0005x^2, & 496.71 \le x < 646.08 \\ 32.724 + 0.0656x - 0.00011x^2, & 646.08 \le x < 790.35 \\ 213.873 - 0.392847x + 0.00018x^2, & 790.35 \le x < 855.44 \\ 740.753 - 1.62468x + 0.0009x^2, & x \ge 855.44 \end{cases}$$

3. LiNbO₃ doped with RuO₂ Concentration 4%

It is very good model since has *adjusted R-Square* of 0.9997, meaning that 99.97% of the percentage of reflectance can be explained by the model. Besides, the t-test has significant intercept, coefficient of x, and coefficient of x^2 .

 $\hat{Y} = \begin{cases} 265.1 - 1.076x + 0.001208x^2, & x < 493.43 \\ -63.5888 + 0.256261x - 0.000142x^2, & 493.43 \le x < 729.50 \\ -819.271 + 2.32804x - 0.001562x^2, & 729.50 \le x < 742.57 \\ 173.268 - 0.345211x + 0.000238x^2, & 742.57 \le x < 860.21 \\ 0.000858x^2 - 1.4119x + 632.04, & x \ge 860.21 \end{cases}$

4. LiNbO₃ doped with RuO₂ Concentration 6%

T-test for all segment in this concentration show that it has significant intercept, coefficient of x, and coefficient of x^2 . The model has *adjusted R-Square* of 0.9998, which is very good because it means 99.98% of the percentage of reflectance can be explained by the model.

$$\hat{Y} = \begin{cases} 110.6 - 0.4063x + 0.00056x^2, & x < 534.77 \\ -207.409 + 0.783028x - 0.000552x^2, & 534.77 \le x < 643.93 \\ -99.6007 + 0.448185 - 0.000292x^2, & 643.93 \le x < 778.59 \\ 258.059 - 0.470551 + 0.000298x^2, & 778.59 \le x < 849.65 \\ 0.001048x^2 - 1.745x + 799.49, & x \ge 849.65 \end{cases}$$

The formed wavelength segment can be studied further by looking for local minimum or maximum points in each segment ($\hat{Y}'=0$). Based on it, a recommendation about maximum and minimum of reflectance for each segmentation could be carried out.



Figure 3: Intersection Of Wavelength Segmentation

Figure 3 shows the regression curve at intersecting wavelengths for all concentrations per segment. In the first segment, the concave curve with the minimum value is produced by LiNbO₃ (0%), which is wavelength (x) of 470.71 with the reflectance of 28.50 percent. The regression curve in the second segment forms a convex pattern, but the optimum point when $\hat{Y}'=0$ is outside the range, because the shape of the curve tends to decrease, so there is no local maximum in this segment, then the optimum point of the curve is the maximum point in LiNbO₃ doped with of RuO₂ concentration of 6%. In the third and fourth segments, there is no intersection with LiNbO₃. The curve is generally convex with the highest local maximum achieved at x = 767.44 with reflectance of 72.38 percent at a concentration of 6%. The curve in the fourth segment is concave with the minimum reflectance percentage of 72,305 reached when the wavelength is at point 789.52 at a concentration of 6%, while the fifth segment, the lowest reflectance is 29.47 percent when the wavelength of 835.79 for concentration of 0%.



Figure 4: The summary of Wavelength Segmentation

In Figure 5, a summary of wavelengths per concentration with a blue dot is a breakpoint between segments of each concentration. In concentration of 0%, the curve in the first segment is concave with a minimum value of x when $\hat{Y}'=0$, which is the percentage of reflectance equal to 25.06 for wavelength 453.53 nm. The second segment is convex but there is no local maximum, while the third segment is convex without a local minimum. The curve in the fourth segment is convex which can rise up to reflectance of 32.66 percent when the wavelength is 663.4 nm, while the fifth segment is concave which can drop to reflectance at 29.47 percent when the wavelength is 835.79 nm. Based on Figure 5 for a concentration of 2% it can be interpreted that in the first segment a concave curve is formed with a local minimum of reflectance of 28.50 percent achieved when the

wavelength is 470.71 nm. The curve in the second and third segments is convex, but there is no local maximum, because the curves that are formed continue to rise. Similarly, in the fourth and fifth segments, the concave shaped curve continues to fall to the segment boundary, consequently there is no local minimum. The same pattern is owned by a concentration of 4%, only the first segment produces the local optimum value from a convex curve when the wavelength is 445.36 nm with reflectance of 25.49 percent. At a concentration of 2% a concave curve is obtained in the first segment with a minimum reflectance of 36.90 for wavelengths at 362.77 nm, the second segment is convex, but there is no local maximum, while the third segment is concave with a local minimum at the wavelength of 767.44 nm with the reflectance percentage is 72.38. The fourth segment curve is concave with a local minimum in the reflectance percentage of 72,305 achieved when the wavelength is at the point 789.52 nm. Concave shaped curves in the fifth segment continue to fall to the upper limit of the segment, so there is no local minimum. The optimum point (local maximum / minimum) when $\hat{Y} = 0$ is illustrated in figure 3, which at 0%, the lowest minimum (concave) peak is produced by first segment with percentage of reflectance = 25.06 for x = 453.53 and the maximum is 32.66 when x = 663.4. Then, the minimum peak point (concave) is in first segment at 28.50 when x = 470.71 concentrations of 2% and 25.49 when x = 445.36 for concentrations of 4%. At a concentration of 6%, the local minimum is in first segment of 36.90 when x = point 362.77 and the maximum is given by third segment which is equal to 72.305 when x =789.52. It is known that the minimum percentage of reflectance is in the first segment and is the highest in general in the fifth segment. If comparing the peak points among concentrations at $\hat{Y}'=0$, reflectance can drop to 25.06 at 0% and maximum up to 72.305 at concentration of 6%.

4. Conclusions

The best model is spline quadratic regression (2^{nd} order) with four knots. The best regression results show the wavelength range per segment for each concentration are different. Adjusted R-Square for each concentration of 0, 2, 4, and 6% are 99.72%, 99.7%, 99.97% and 99.98% respectively. The significance wavelength segment in this research is very useful for processing advanced material data to develop LiNbO₃ and its technology in the future. Recommendations related to the local peak point of each segment obtained that at the intersection of wavelength in the first segment, the minimum reflectance is produced by LiNbO3. In the second and third segment slices, the maximum reflectance produced by the concentration of 6%, while the fourth segment, the minimum percentage of reflectance is reached by a concentration of 6%. In segment 5, based on the same intersection, the minimum reflectance can be recommended by LiNbO₃. Based on the concentration, at a concentration of 0% the minimum reflectance was obtained at 25.06 percent at a wavelength of 453.53 nm and a maximum of 32.66 percent when the wavelength was 663.4 nm, while at a concentration of 2% and 4% only had the minimum reflectance of 28.50 and 25.49 percent. The recommendation for a concentration of 6% is the minimum point can be obtained when the reflectance is 36.90 percent when the wavelength is 362.77 nm and the maximum is reached at 72.305 percent when the wavelength is 789.52 nm.

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