



QUANTIFICATION OF DICYANDIAMIDE IN MILK PRODUCTS USING FTIR SPECTROSCOPY METHOD

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Introduction

Dicyandiamide, commonly known as DCD or Cyanoguanidine, is a guanidine that has one of its amino hydrogens substituted by a cyano group. Mainly DCD is used by dairy farms to reduce greenhouse gas emissions and nitrate leaching into rivers and other waterways from the pasture. Because DCD can act as a nitrification inhibitor.

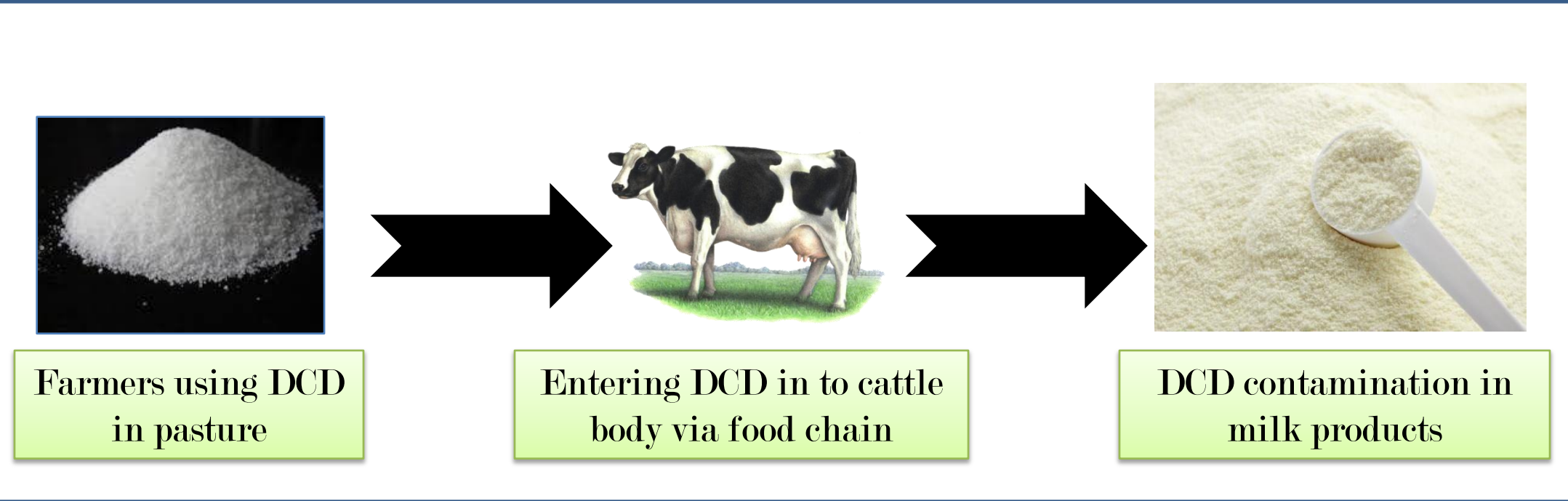


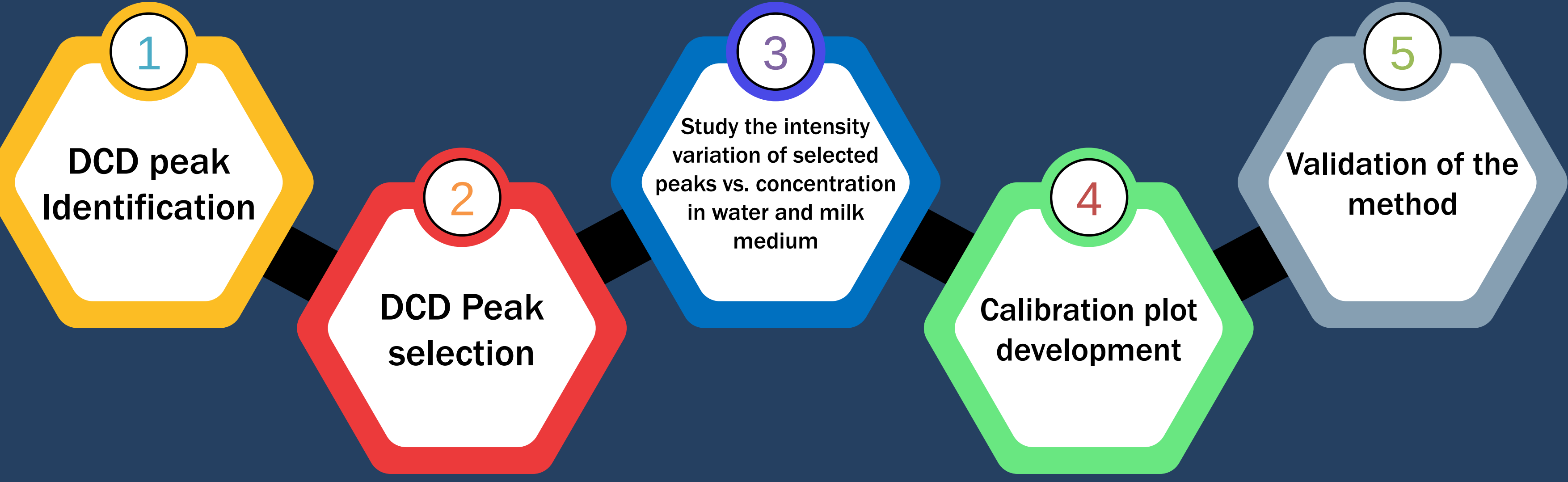
Figure 1: DCD contamination of milk products



Objective

To establish a rapid, efficient and low cost analytical method to detect and quantify the amount of Dicyandiamide adulterant in milk products by using Fourier-transform infrared spectroscopy.

Methodology



Results and Discussion

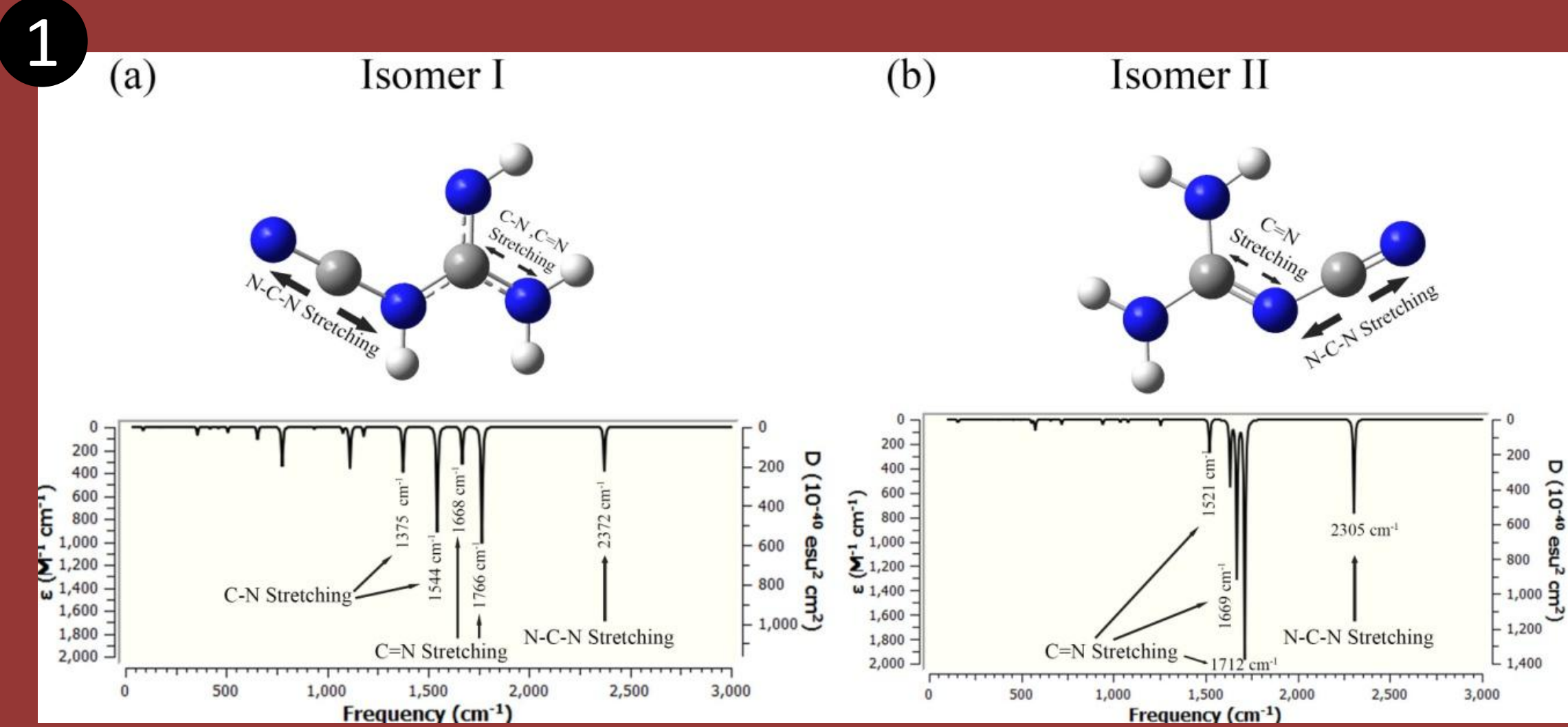


Figure 2: Calculated IR spectra of DCD isomers
•N-C-N stretching was responsible for the peaks at 2200 cm⁻¹ and 2160 cm⁻¹.

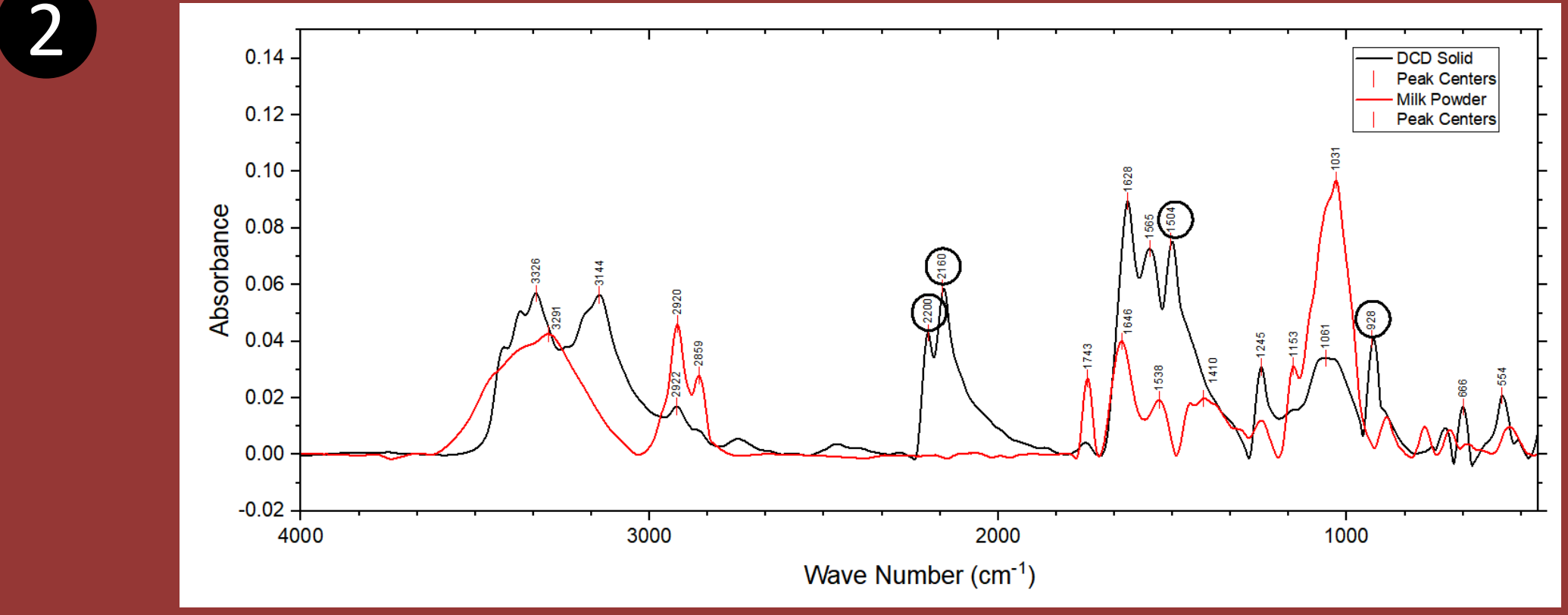


Figure 3: Superimposed DCD and milk FTIR spectra
•After the visual inspection, Peaks at 2200, 2160 1504, and 928 cm⁻¹ wave numbers were selected as the potential peaks for calibration.

Concentration series	R ² values for selected peaks in water concentration series			
	928 cm ⁻¹	1504 cm ⁻¹	2160 cm ⁻¹	2200 cm ⁻¹
0.01-0.1 mg/ml	0.38	0.04	0.84	-0.1
0.1-1 mg/ml	-0.07	0.79	0.53	0.83
1-10 mg/ml	0.92	0.67	0.92	0.97

Table 1: Linearity analysis of DCD concentration series with water

•1-10 mg/ml DCD concentration series with both water and milk showed good linearity for selected peaks.

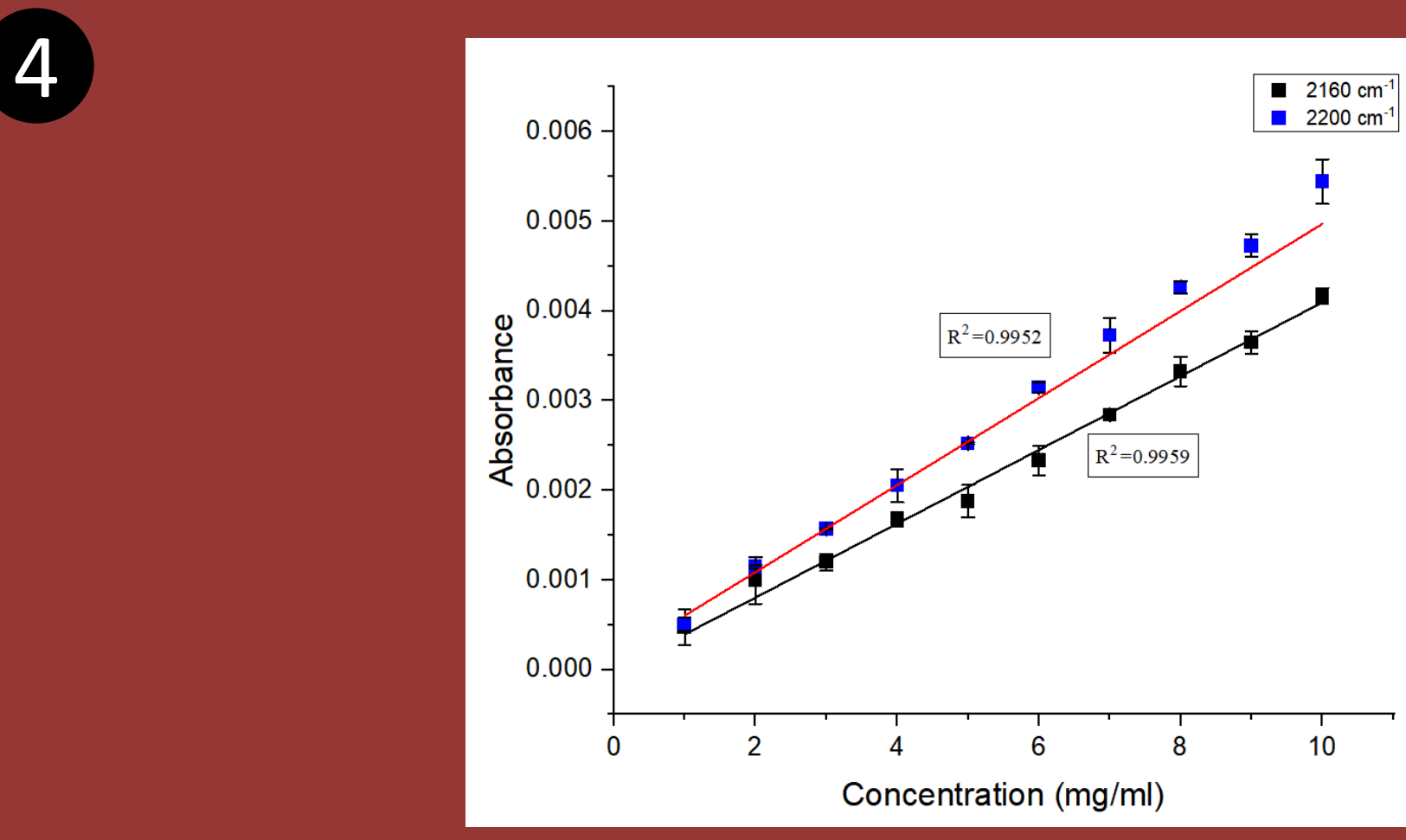


Figure 4: Calibration plots for peak intensities of 2200 and 2160 cm⁻¹

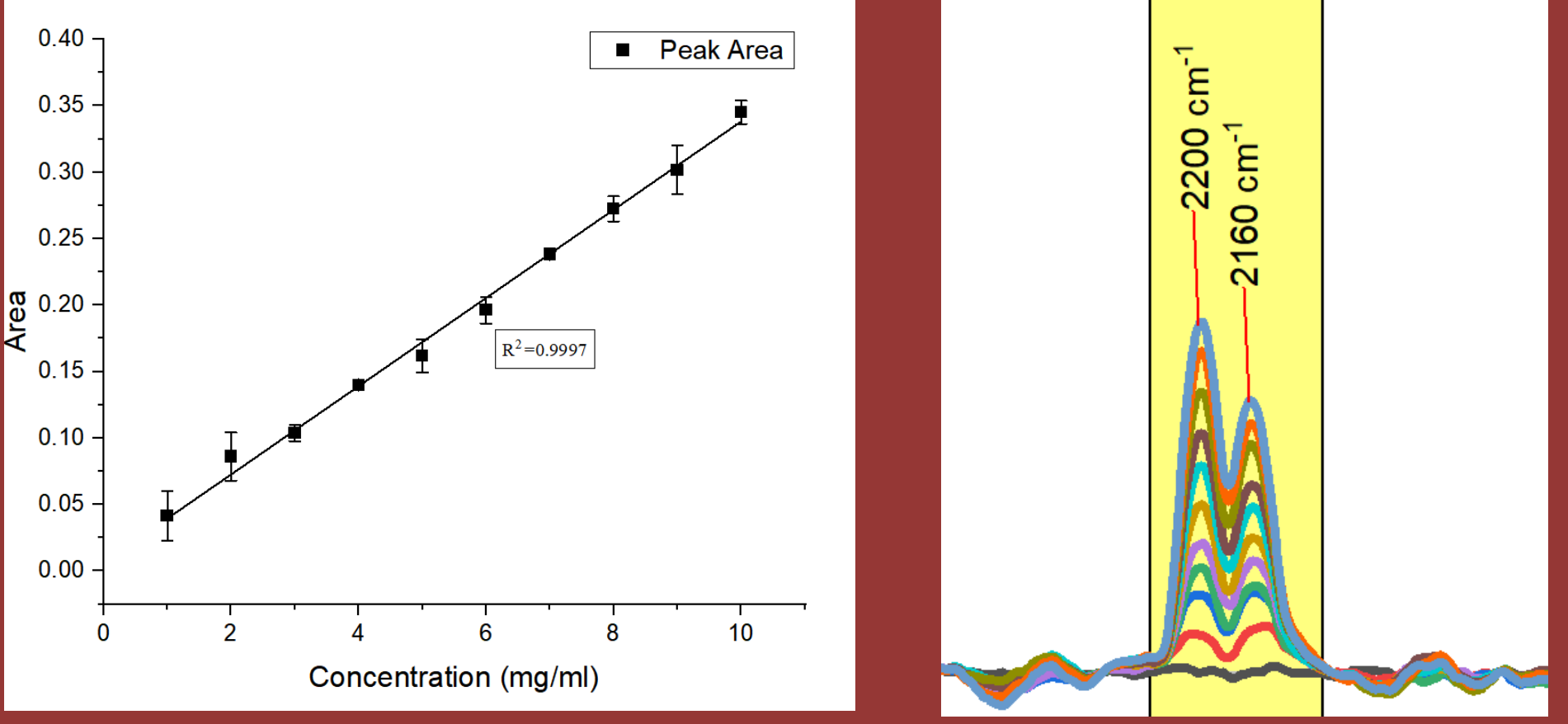


Figure 5: Calibration plots for peak area of 2200 and 2160 cm⁻¹

•2200 and 2160 cm⁻¹ peaks were chosen to develop calibration plots of DCD quantification and the calibration plots have linear fit with R² values greater than 0.9.

Plot	LOD (mg/ml)	LOQ (mg/ml)
2160 cm-1	0.49	1.48
2200 cm-1	0.85	2.58
Peak area	0.74	2.23

Table 2: Detection and quantification limits of calibration plots

•The validation results gave mean recovery percentages of 99 ±7.69% (2160 cm⁻¹), 105 ±3.97% (2200cm⁻¹), and 113 ±18.71% (peak area) calibration plots.

Conclusions

- Four potential Peaks at 2200, 2160 1504, and 928 cm⁻¹ wave numbers were selected as the potential peaks.
- 2200, 2160 cm⁻¹ peaks were responsible for the N-C-N stretching of both tautomers present in DCD.
- Only 1-10 mg/ml DCD concentration series with both milk and water showed good linearity with selected peaks.
- 2200 and 2160 cm⁻¹ peaks were chosen for the calibration plot development
- This work gave a minimum LOD of 0.49 mg/ml and a LOQ of 1.48 mg/ml for a 2160 cm⁻¹ calibration plot.
- * Future works will have to address on improving sensitivity of selected FTIR peaks to reduce the LOD values.

References

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(2) Lin, X.; Hasi, W. L. J.; Lou, X. T.; Han, S. Q. G. W.; Lin, D. Y.; Lu, Z. W. Direct and Quantitative Detection of Dicyandiamide (DCD) in Milk Using Surface-Enhanced Raman Spectroscopy. *Anal. Methods* **2015**, 7 (9), 3869–3875.

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