OPTIMIZATION OF CONDITIONS FOR THE SEPARATION OF SUGAR COMPOUNDS EXTRACTED FROM $PM_{2.5}$ SAMPLES USING ION CHROMATOGRAPHY WITH PULSED AMPEROMETRIC DETECTION

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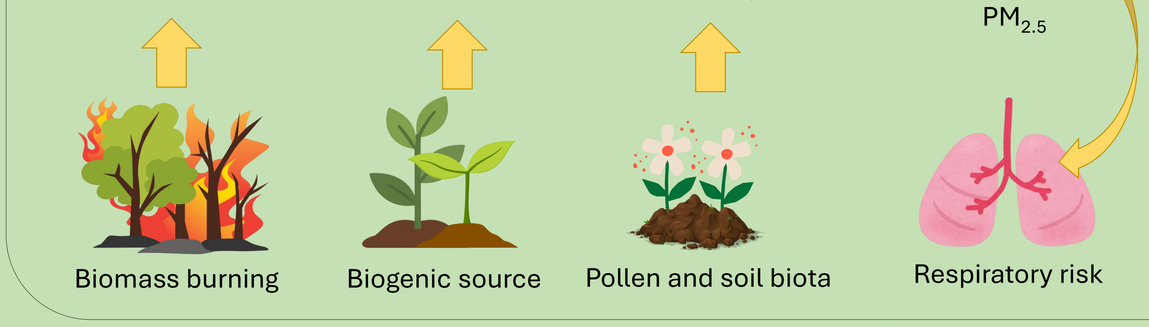
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ABSTRACT Sugar compounds are commonly present in fine particulate matter ($PM_{2.5}$). In this study, sugars were determined using Ion Chromatography coupled with Pulsed Amperometric Detection (IC-PAD), an established analytical technique for carbohydrate analysis. The method focused on the separation and optimization of sugar compounds by adjusting the elution conditions, the initial sodium hydroxide (NaOH) concentration, and the column temperature. Optimal separation was achieved using stepwise gradient elution with 150 mM sodium hydroxide at 40 °C. The method demonstrated good linearity (0.01 – 10.00 mg L^{-1}), low method detection limit (MDL, 0.002 – 0.029 mg L^{-1}), limit of quantitation (LOQ, 0.005 – 0.088 mg L^{-1}), high precision (RSD < 1.1%), and good accuracy (recoveries 90.5 – 104.3%). The improved method was shown to be efficient enough to quantification of sugars in $PM_{2.5}$ samples from Chiang Mai province, Thailand.

INTRODUCTION

Sources of sugar compound in $PM_{2.5}$



IC-PAD for sugars analysis

IC-PAD enables sugar analysis under strongly basic conditions (pH > 12), where sugars are converted to oxyanions, separated by anion-exchange chromatography, and detected by pulsed amperometry to generate chromatograms.

Co-elution of Critical Sugars Pairs

Co-elution of critical sugar pairs, particularly Levoglucosan/Arabitol and Mannosan/Sorbitol, limits accurate quantification of these analytes.

OBJECTIVE

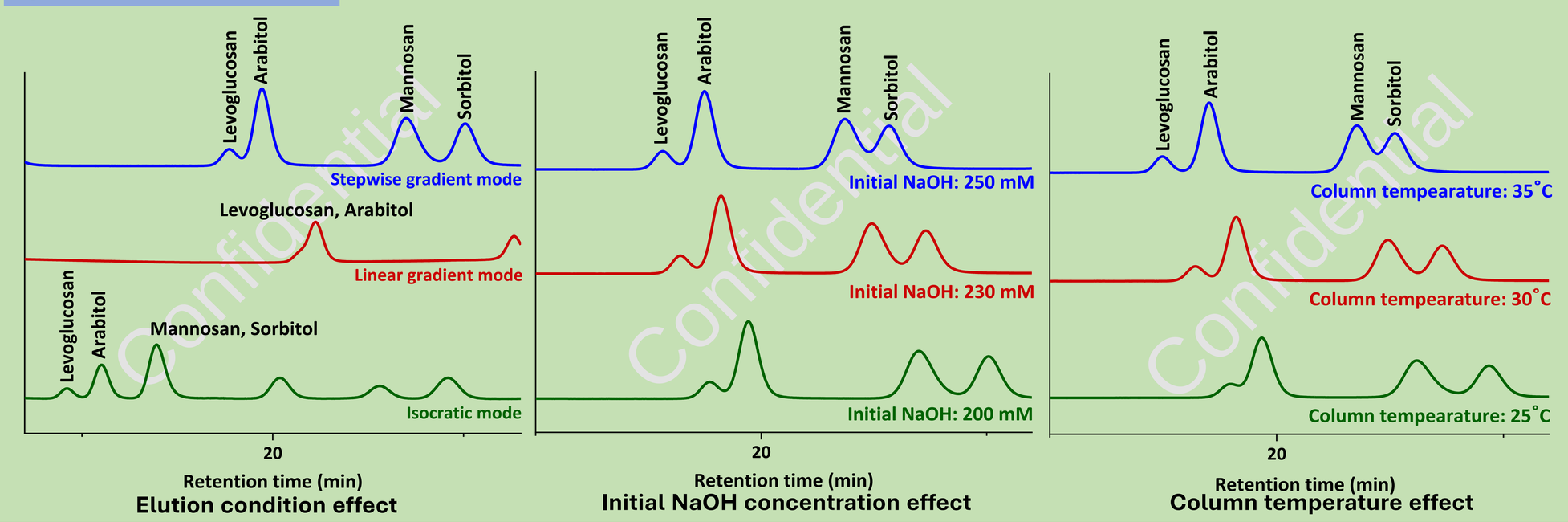
To develop an IC-PAD method for the analysis of sugars commonly found in $PM_{2.5}$ samples and to resolve the co-elution of Levoglucosan/Arabitol and Mannosan/Sorbitol.

METHODS

Separation conditions for 12 standard sugars were optimized by adjusting elution conditions, initial NaOH concentration, and column temperature. The analysis was performed using IC-PAD with NaOH as the eluent.

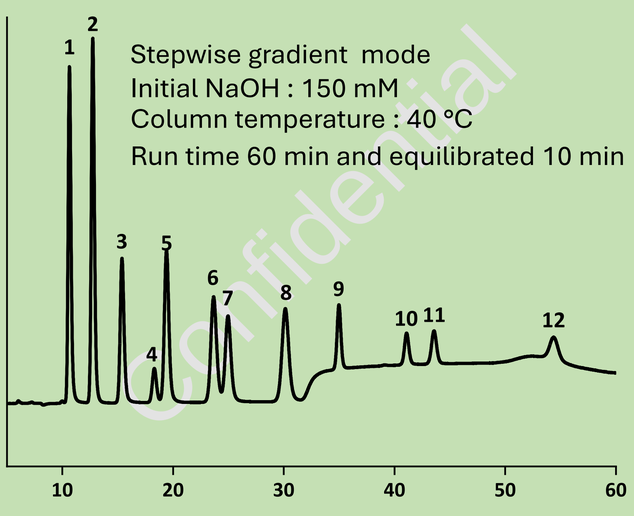
RESULT AND DISCUSSION (UNPUBLISHED)

Optimization of Conditions



- 12 sugars were analyzed; only the two most difficult pairs are shown
- Stepwise gradient mode can be separate both sugar pairs (Levoglucosan/Arabitol, LG/AR) and (Mannosan/Sorbitol , MN/SB)
- Higher NaOH concentration and column temperature increased resolution of LG/AR but decreased resolution MN/SB

Optimal conditions



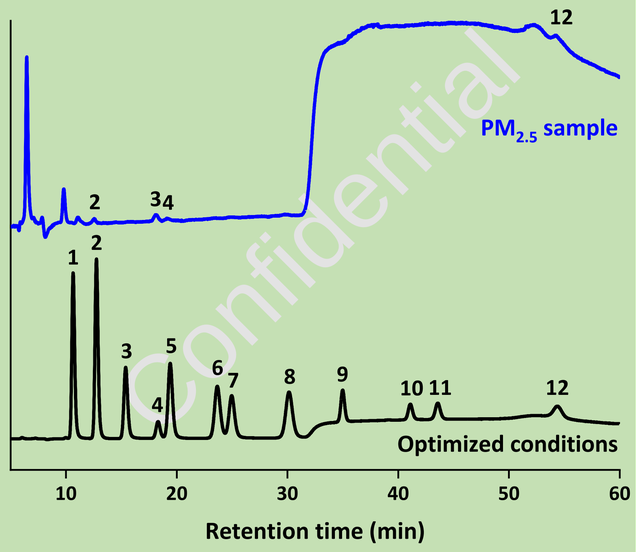
Method Validation

Sugar	Calibration curves		% recovery	%RSD of concentration		%RSD of retention time		MDL	LOQ
	Range (mg L^{-1})	R^2		intra-day	inter-day	intra-day	inter-day	(mg L^{-1})	(mg L^{-1})
Inositol	0.01-10.00	≥0.999	96.4-97.7	0.25	0.82	0.16	0.03	0.009	0.027
Erythritol	0.01-10.00		94.9-99.2	0.22	0.93	0.13	0.02	0.006	0.019
Xylitol	0.01-10.00		96.4-99.4	0.17	0.77	0.20	0.03	0.010	0.032
Levoglucosan	0.01-10.00		97.8-102.4	0.33	0.28	0.24	0.04	0.005	0.015
Arabitol	0.02-10.00		97.5-100.2	0.24	0.94	0.24	0.03	0.002	0.005
Mannosan	0.04-10.00		95.5-99.2	0.28	1.11	0.32	0.03	0.009	0.028
Sorbitol	0.01-10.00		96.5-98.7	0.32	1.13	0.40	0.04	0.004	0.013
Mannitol	0.04-10.00		90.9-97.5	0.30	0.73	0.38	0.03	0.009	0.028
Galactosan	0.04-10.00		97.8-99.9	0.36	0.41	0.25	0.05	0.013	0.040
Glucose	0.04-10.00		90.5-96.8	0.93	0.73	0.30	0.06	0.025	0.075
Galactose	0.04-10.00		92.3-96.5	0.89	0.64	0.31	0.05	0.029	0.088
Sucrose	0.31-10.00		92.5-104.3	0.46	0.28	0.34	0.05	0.024	0.072
Range			**90.5 – 104.3**	**0.17 – 0.93**	**0.28 – 1.13**	**0.13 –0.40**	**0.02 – 0.06**	**0.002 – 0.029**	**0.005 – 0.088**

Good linearity | Good accuracy | High precision | Suitable MDL and LOQ

"Method validation that confirming its reliability for sugar analysis in $PM_{2.5}$ samples"

Sugar analysis in $PM_{2.5}$ samples



Sugars

1- Inositol
2- Erythritol
3- Xylitol
4- Levoglucosan
5- Arabitol
6- Mannosan
7- Sorbitol
8- Mannitol
9- Galactosan
10- Glucose
11- Galactose
12- Sucrose

CONCLUSION

The optimized IC-PAD method resolved critical co-elutions, complied with validation criteria, and was successfully applied to real $PM_{2.5}$ samples from Chiang Mai province, Thailand.

REFERENCES

1. Patil, S., & Rohrer, J. (2019). Determination of saccharides in atmospheric aerosols using high-performance anion-exchange chromatography with pulsed amperometric detection (HPAE-PAD) (Application Note 73009). Thermo Fisher Scientific.
2. Patil, S., & Rohrer, J. (2022). A fast, single column high-performance anion exchange chromatography with pulsed amperometric detection method for the determination of saccharides in atmospheric aerosol samples. Journal of Separation Science, 45(18), 3422-3430.

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