

# **Exploring the Differences of Beet and Cane Sugar through NMR Relaxometry**

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### Abstract

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Table sugar, chemically known as sucrose, is one of the purest organic food products in the world with the purity of 99.9%. It is the main ingredient of almost each food products which is sweet in taste and extracted either from sugar beet or sugar cane in all over the world. The cost of production of cane sugar is significantly lower than beet sugar which ultimately leads to the unjust competition in the markets. Since both beet sugar and cane sugar are almost similar in physical as well as chemical properties, it is really difficult to differentiate



between them. In this study, 15 different sugar samples obtained from different countries were tested using TD-NMR Relaxometry; FFC-NMR Relaxometry and X-ray diffraction analysis.  $T_1$  and Solid Echo measurements were conducted in 20.34 MHz (<sup>1</sup>H) system. <sup>1</sup>H spinlattice relaxation measurements have been performed for a series of mixture of sugar and water in the frequency range of 10kHz-10MHz in 25°C, using Smartracer FFC-NMR relaxometer (Stelar s.r.l.).  $T_1$  and SE measurements at permanent field did not show any significant difference (p>0.05) among the samples. Examples of NMRD profiles for white (Sugarbeet, Südzucker) and brown (Sugarcane, Hellma) Sugar are shown in Figure 1 (a). The relaxation processes has turned out to be biexponential in the whole frequency range for all sugars as shown in Figure 1 (b). The averaged value of the ratio between the amplitudes of the fast and slow relaxation contributions are about 3.0 (white sugar) and 4.4 (brown sugar). XRD results also showed significant differences among the samples. Crystallinity and crystallite size were obtained from the measurements. The maximum 88.55% and minimum 70.33% of crystallinity in cane sugar while the maximum 88.90% and minimum 71.01% of crystallinity in beet sugar was observed. In case of crystallite size, the maximum 39.41 nm and minimum 24.68 nm of size of crystallite in cane sugar whereas the maximum 42.58 nm and minimum 25.0 nm in beet sugar was noticed.

Figure 1: a; NMRD profiles, B; Biexponential <sup>1</sup>H spin-lattice relaxation of beet sugar and cane sugars.

### Introduction

Sucrose, is non-oxidative disaccharides produce by plants as a transportable material in nature. It exists in three physical forms crystalline, amorphous, and liquid. Although it provides i.e. various functional properties to food like sweetness, aroma, nutritional value, texture, color, caramelization, preservation, fermentation and antioxidant properties but mainly it is consumed a lot by human beings because of its sweetness and nutritional values. Sucrose can be produced from several sources but in



### Results

T1 and SE measurements at permanent field did not show any significant difference (p>0.05) among the sugar samples so unable to divide into groups based on the sources of sugars. The 1H spin relaxation was biexponential in the whole frequency range for all sugar samples (Figure 2). The results indicated the clear differences in 1H spin relaxation rate for all sugars supported by XRD results. The differences in averaged value of the ratio between the amplitudes of the fast and slow relaxation were observed about 3.0 in beet sugar whereas 4.4 in cane sugar with some exceptions i.e. The cane sugar sample, ITWC (D), aligned with beet sugar in results.

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almost similar in physical as well as chemical properties, it is really difficult to differentiate between them. In this study, the differences between 15 sugar samples obtained from different induestries worldwide were tested using TD-NMR Relaxometry; FFC-NMR Relaxometry and X-ray diffraction analysis.

## **Materials and Methods**

The 15 sugar samples were taken from the different sugar Brands/industries of 8 different countries and coded them (Table 1). The 6 samples were from beet sugar and 6 samples were from cane sugar while the source of 1 sample was unknown.

 $T_1$  and *Solid Echo* measurements were conducted in 20.34 MHz (<sup>1</sup>H) system (TD-NMR).

The X-ray diffraction of samples were studied by using Rigaku Ultima-IV X-ray diffractometer at METU Central Laboratory with the scan range of  $0^{\circ}$ – 70°. The crystallinity and crystallite size of samples were calculated.

<sup>1</sup>H spin-lattice relaxation measurements were performed for 50% solution (w/w) in the frequency range of 10kHz-10MHz at 25°C, using Smartracer FFC-NMR relaxometer (Stelar s.r.l., Italy).

Table 1: The list of sugar samples with code,

locality, sugar types, sugar source.

Sr. No.	Country	Code	Sugar Type	Source
1	Germany	DEBC (H)	Brown Sugar	Sugarcane
2		DEWB (Su)	White Sugar	Sugarbeet
3	Italy	ITBC (D)	Brown Sugar	Sugarcane
4		ITWC (D)	White Sugar	Sugarcane
5	Pakistan	PKWC (SG)	White Sugar	Sugarcane
6	Poland	PLWB (AE)	White Sugar	Sugarbeet
7		PLWB (D)	White Sugar	Sugarbeet
8		PLWB (SC)	White Sugar	Sugarbeet
9	Portugal	PTBC (C)	Brown Sugar	Sugarcane
10		PTWC (S)	White Sugar	Sugarcane
11	Romania	ROW- (MZ)	White Sugar	Unknown
12	Serbia	RSWB (C)	White Sugar	Sugarbeet
13		RSWB (DO)	White Sugar	Sugarbeet
14		RSWB (S)	White Sugar	Sugarbeet
15	Turkey	TRWB (KS)	White Sugar	Sugarbeet



#### Figure 2: Biexponential <sup>1</sup>H spin-lattice relaxation of all sugar samples.

XRD results also showed significant differences among the sugar samples (Figure 3). The maximum 88.55% and minimum 70.33% of crystallinity in cane sugar while the maximum 88.90% and minimum 71.01% of 21500000 crystallinity in beet sugar was observed (Figure 4a). In case of crystallite size, the maximum 39.41 nm and minimum 24.68 nm of size of crystallite in cane sugar whereas the maximum 42.58 nm and minimum 25.0 nm in beet sugar was noticed (Figure 4b). Hence, XRD can be used to differentiate between the









Figure 2: a; Graph showing the percentage of Crystallinity, b; Graph showing the Crystallites size. The column in brown color representing the brown sugar.

sugars of different brands but not enough for the sources.

#### **Figure 3: X-ray Diffraction spectra of** sugar samples.

# Conclusion

The current study revealed that the FFC-NMR coupled with XRD is good techniques to differentiate between the sugars produced by different brands or industries. The variation between beet sugar and cane sugar was found in the results of FFC–NMR spin relaxometry measurements with some exceptions of cane sugar which showed the behaviour like beet sugar in the results. Since, the limited number of sugar samples were analysed in this study, so the more details study on large number of sugar samples is required to say that FFC–NMR spin relaxometry can be used to differentiate between the sugars of different sources.

#### References

1. Asadi, M., Beet-sugar handbook. 2007: John Wiley & Sons.

2. Eggleston, G., Sucrose and related oligosaccharides. Glycoscience, 2008: p. 1163.



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