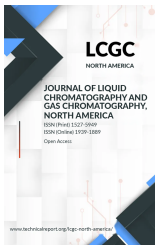

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RESEARCH ARTICLE

Mode-Specific Food-Sugar Information Using NiFe Nanowire Electrodes: Class Project, Dilution Positioning, and Recovery/Dispersion Analysis

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Abstract

For food-sugar electroanalysis, it is important to have a chemically meaningful value of the concentration, not just an elevated electrode current. This paper addresses the research question on the viability of developing a mode-specific rule for food-sugar readings by NiFe alloy nanowires using chronoamperometry on five different reducing sugars and seven food samples. The analytical system contains glucose, fructose, galactose, lactose, and maltose calibration information; direct reducing-sugar measurements for peach juice, honey, diet coke, milk, isotonic solution, and apple; and total-sugar measurements for peach juice, coke, diet coke, and apple, including conversion from non-reducing sugar. Calculations include calibration window usefulness, carbon-normalised response density, cross-family concentration difference, recovery agreement, replicate dispersion, and liquid sample dilution position. Glucose had the highest usefulness among single sugars, whereas fructose and galactose had an informative monosaccharide family and lactose/maltose had a less responsive disaccharide family. Monosaccharide mean sensitivity was 0.6427 μA μM^{-1} cm^{-2} and disaccharide mean sensitivity was 0.3555 μA μM^{-1} cm^{-2}, with a raw slope ratio of 1.81. The use of carbon-normalised response density amplified the class difference to 3.62. This demonstrates that the disaccharide response is not proportional to the molecule size. Cross-family slope transfer underestimates disaccharides by 44.7% or overestimates monosaccharides by 80.8%. The average recovery of direct reducing-sugar measurements was 98.05%, the average recovery of total-sugar measurements was 99.11%, and non-zero replicate dispersion was lower in direct measurements than in total-sugar measurements. Liquid foods needed approximately 854–4225-fold dilution to bring the aliquot close to 0.18 mM within the common first calibration window. The research question is answered positively: NiFe nanowire food-sugar readings are best expressed by mode chemistry, with class-specific calibration, interior window dilution, and recovery-dispersion interpretation mentioned together.

Keywords: NiFe nanowires, reducing sugars, total sugar, non-enzymatic sensing, chronoamperometry, food analysis, dilution placement, apparent recovery

1. Introduction

The problem of sugar measurement in food is a chemical measurement rather than a standard calibration task for a specific compound. Nutritional labelling, sweetness control, fermentation monitoring, ingredient validation, and batch release all depend on whether the measured concentration is related to reducing sugars, total sugars, added sugars, free sugars, or a specific equivalent of sugar content in the particular matrix. The same food sample contains glucose, fructose, galactose, lactose, maltose, sucrose, polyols, organic acids, salts, proteins, pigments, and carbonated products. Therefore, a good electrochemical signal in case of glucose measurement is not sufficient to declare va-

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lidity in case of milk, honey, fruit juice, or any beverage that contains non-reducing sucrose. The crucial issue is whether the NiFe nanowire-based measurement of food-sugar concentration can be transferred into a chemical-assignment approach.

The differentiation of total, added, free, and reducing sugars is important because analytical definitions are not always identical to those in nutritional statements and vice versa. Nutritional considerations demonstrate that the health implications of sugar are dependent on the declaration rather than on the molecules themselves [1]. Estimation of added sugars in packaged foods demonstrates the inability to convert total-sugar declarations into added-sugar concentrations without product-related assumptions [2]. The electrochemical sensors face similar challenges. Direct anodic current in alkaline electrolyte defines electroactive reducing species under the selected experimental conditions; the total sugar after the acidification and heating procedure defines a different pool of the reduced sugars including the converted sucrose. This difference should be clearly stated in the analysis description and not hidden in a footnote.

Classical methods of food-sugar determination are still relevant because they precisely identify the analyte. Reducing sugar titration and Benedict test provide chemically simple operational approaches, and the recently developed quantitative analysis demonstrates their applicability for the determination of measurable reducing sugars [3]. The higher composition resolution is provided by the chromatographic technique: high-performance liquid chromatography with post-column detection is used for the determination of reducing sugars in the food matrix [4]. Electrochemical flow-injection analysis was applied for fast reduction of sugars in potatoes as well as other foods; short analysis time is beneficial in the case when the target pool is identified [5]. Electrochemical analysis of honey with a nickel complex sensor and chemometric interpretation demonstrates that sensor response can be beneficial for complex foods if matrix-related processing is considered [6]. Simultaneous monitoring of sucrose, glucose, and fructose in flow system demonstrates that total-sugar and reducing-sugar determination requires combination of the chemical and electrochemical operations rather than one unqualified current response [7].

The most recent reviews of electrochemical sensors in the field of food analysis emphasize fast analysis time, low instrumental complexity, and the wide usage of nanostructured electrode materials in routine food-screening applications [8]. This approach is attractive in case of sugar-rich samples when the fast screening could be accompanied by more precise reference technique. The same literature also indicates why the instrumental performance cannot be evaluated solely by detection limits. Electrode materials, surface chemistry, selectivity, matrix compatibility, and calibration design are all the factors that affect sensor applicability from a model solution to the real product. In case of sugars that are present in foods at grams-per-litre level, the initial challenge is not the presence of trace amounts in the undiluted sample but the ability to prepare diluted aliquot inside a reliable calibration interval.

Glucose-sensor development in the clinical monitoring field is characterized by glucose as a target analyte and the defined matrix. Early reviews on electrochemical glucose biosensors identified the importance of the enzyme immobilization, mediator chemistry, electrode design, and sensor stability [9, 10]. More recent surveys of enzymatic and non-enzymatic glucose sensors describe the role of nanomaterials, metal oxides, carbon materials, and hybrid electrode in improving current response and stability [11, 12]. This information explains the benefits of non-enzymatic glucose sensors but does not reflect the requirements of food analysis when the target could be sugar class rather than glucose molecule, and the matrix could be juice, milk, honey, or carbonated beverage.

The non-enzymatic sensors are useful because of the absence of storage limitations and the possibility of using direct electrocatalytic sugar oxidation in alkaline media. The reviews on non-enzymatic glucose sensors highlight transition-metal surfaces and metal oxides as the sources of improved electrocatalysis [13, 14]. Various types of alloy and composite electrodes were also investigated [15, 16]. More recent reviews emphasize porous structures and engineering of interfaces [17, 18]. Nickel-based materials are important because of the involvement of nickel hydroxide/oxyhydroxide transformation in carbohydrate oxidation in alkaline media. The design of the nickel-rich surface is defined by the accessibility of the active sites, redox state, and morphology of nanostructure [19]. Iron-containing nickel materials are also important because Ni–Fe interactions could change the electrochemical properties of electrodes. NiFe alloys and layered-hydroxide materials are the examples of this approach [20, 21]. The similar effect is also demonstrated in other NiFe hybrid materials and compositionally tunable structures [22, 23]. The charge-transfer modifications are discussed for NiFe systems as well [24].

The food-chemistry requirements are not limited to the good current response of glucose. Glucose, fructose, and galactose are C6 monosaccharides, whereas lactose and maltose are C12 disaccharides. Milk is rich in lactose, fruit and many beverages contain reducing monosaccharide fraction, and sucrose-containing foods require preliminary conversion for total-sugar determination. Sugar-sensing studies show that non-enzymatic sensors can respond to various sugars. Nickel–chromium alloy and nickel nanoflowers are the examples of such electrodes [25, 26]. Copper–nickel system extends the response [27, 28]. Renewable copper-based electrodes are also available [29]. The ability to respond to multiple sugars is not equal to the universal calibration because the slope for each sugar is specific and the calculated concentration would differ depending on the selected slope.

Therefore, the NiFe nanowire food-sugar analysis is a chemical-assignment problem based on three related decisions: sugar-class assignment, dilution position, and recovery-dispersion interpretation. The numeric basis of the research consists of five rows with sugar-level calibration and food-sample rows with peach juice, honey, coke, diet coke, milk, isotonic solution, and apple. The same NiFe nanowire chronoamperometric measurements are interpreted based on the chemical nature of sugar pool rather than on the glucose-only rankings. The research question is whether the record could provide the rule of food quality that guarantees the lack of calibration-class error and separation of total-sugar and reducing-sugar concentrations.

The dilution rule is especially important because several food samples are expressed in g/L. The sample that contains tens or hundreds grams per liter of sugar cannot be analyzed with micromolar calibration line. It should be diluted in order to be able to measure chronoamperometric current. Under such conditions, lower LOD is not so important as the stable slope, the correct calibration class, and the reproducibility of placement inside the first linear interval. The internal target used here is 0.18 mM, selected within the common 0.05–0.30 mM first interval and over the LOQ values of all five sugars. This target defines a practical point where various liquid matrices can be compared without forcing them to the edge of the calibration interval.

The analysis includes calibration window utility, carbon-normalized response density, class-transfer bias, apparent recovery, replicate dispersion, and liquid dilution. Each table and figure reflects the corresponding numerical result and its chemical role in the food-sugar determination. Conclusion comes back to the research question and gives the answer: NiFe nanowire analysis is most acceptable for food-quality applications when the declared concentration specifies chemical mode, calibration class, dilution position, and recovery-dispersion situation.

2. Materials and Methods

2.1. Measurement Basis and Analytical Scope

The analysis takes the sugar levels of the NiFe nanowires from [30]. Sugar-levels include the first linear interval, sensitivity, repeatability,

reproducibility, limit of detection, and limit of quantification of glucose, fructose, galactose, lactose, and maltose. Food-levels include the determination of the direct reducing sugar and the total sugar of peach juice, honey, coke, diet coke, milk, isotonic solution, and apple, along with the corresponding titration, label, and literature reference value where possible. Numerical analysis categorizes the values according to sugar class, dilution step, apparent recovery, and replicate scatter. Figure 1 shows the material and measurement context of these values by connecting the ordered NiFe nanowire surface to the alkaline three-electrode cell for chronoamperometric sugar level readings.

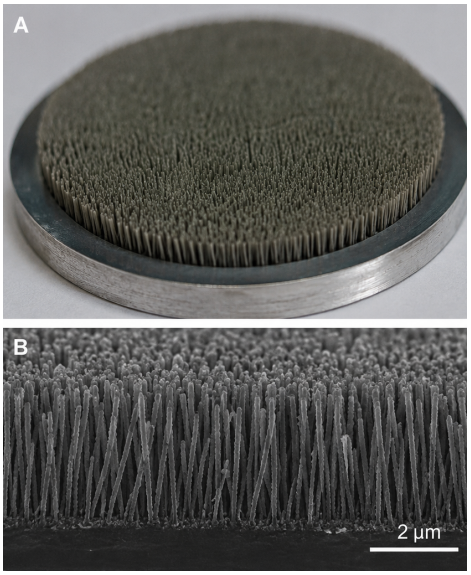


Figure 1: NiFe nanowire electrode and alkaline chronoamperometric cell.

The material context picture is important since the numerical values rely on a particular electrode arrangement rather than an abstract current response. The nanowire provides the catalyst surface, whereas the alkaline cell fixes the electrolyte and the potential environment in which the sugar currents have been determined. Interpretation of the numerical values without such a context would obscure the reason for the importance of dilution, potential control, and surface access in this method.

The sugar-level data sheet includes the parameters needed for the calculations in Table 1. The five sugars have one common first linear interval, namely 0.05–0.30 mM. Glucose, fructose, and galactose are monosaccharides; lactose and maltose are disaccharides. The classification is chemical and not statistical since it refers to the molecular size, number of carbons, and the food sources in which these sugars typically appear. The common upper bound of the first linear interval is designated $C_U = 300\ \mu$M.

The above calibration data form the limit of analysis of the study. The common 0.05–0.30 mM range allows for a reasonable comparison between classes, whereas the differences in sensitivity and quantification limits prove that a common range does not imply a common slope. The table thus provides the first answer to the research question: class determination needs to precede concentration assignment.

The food-level values are presented in Table 2. RS stands for reducing sugars, T-RS stands for total reducing sugars after treatment, SM stands for standard method, TT stands for titration, LB stands for label value, Ref stands for literature reference value, and NT stands for not tested. Unit conservation in this table is required due to its necessity for dilution assignment. G/L samples can be assigned molar concentration after selection of the representative molar mass. Samples given per gram of foodstuff, such as honey and apple, cannot be directly translated into molar concentration without information on density/solid sample preparation volume; hence they are kept in recovery/dispersion calculation rather than in g/L dilution table.

The foods chosen provide a deliberately heterogeneous validation set. Peach juice and isotonic solution provide liquid samples that need extensive dilution; milk provides a disaccharide (lactose) sample; honey and apple are mass-normalised samples; coke and peach juice sugar entries depend on conversion for total-sugar determination; while diet coke is a zero sugar control. It is due to this heterogeneity that results cannot be considered as a simple glucose sensor example.

2.2. Utility of Calibration-Window Approach

Utility of Calibration-window approach allowed to consider slope, detection limit, quantification limit, and repeatability-reproducibility penalty simultaneously within the sugar level indicator. For sugar i, the following notations are used: sensitivity - S_i, detection limit - LOD_i, quantification limit - LOQ_i, repeatability - R_i, reproducibility - P_i, the upper boundary of the first interval - $C_U = 300\ \mu$M. The utility value was

$$U_i = \frac{S_i}{LOD_i} \cdot \frac{C_U}{LOQ_i} \cdot \frac{1}{1+(R_i+P_i)/100}. \tag{1}$$

The normalised utility value was calculated as

$$U_i^* = 100\frac{U_i}{\max(U_i)}. \tag{2}$$

This is not an alternative to the validation process of the method itself. This is a concise way of expressing the comparative performance of the sugar-specific application of the first calibration interval. The first coefficient rewards a high current response value compared with the detection limit. The second one rewards a low quantification cut-off value compared to the upper limit of the common interval. The third coefficient punishes a high penalty on repeatability and reproducibility factors. Thus, this scoring system allows the ranking of sugar calibrations but does not allow food application without class assignment and dilution.

2.3. Molar-Carbon Response Density

The sensitivity values were also normalized in respect to carbon number to examine if class separation is due to different sizes of molecules. The carbon number of the sugar molecules was designated as $n_{C,i}$. For glucose, fructose, and galactose $n_C = 6$, while for lactose and maltose $n_C = 12$. The molar-carbon response density was calculated as

$$D_i = \frac{S_i}{n_{C,i}}. \tag{3}$$

Class means were calculated as

$$\bar{D}_c = \frac{1}{n_c}\sum_{i\in c} D_i, \tag{4}$$

where c is either the monosaccharide or disaccharide class. The molar-carbon class contrast was

$$\Psi_D = \frac{\bar{D}_{mono}}{\bar{D}_{di}}. \tag{5}$$

This normalisation does not seek to suggest that each carbon is oxidised in a way that is equally probable. This is a descriptor of deployment. In cases where the response varies proportionately with molecular carbon number, the per-carbon concentration in classes will be nearly the same. A significant discrepancy of D_i means that response of disaccharides is influenced by more factors than just the formula size, namely, diffusion, orientation, adsorption, hydroxyl availability, and surface redox chemistry.

Table 1: Sugar-level NiFe nanowire performance values used for class-resolved analysis.

Sugar	Linear range (mM)	Sensitivity (μA μM^{-1} cm^{-2})	Repeatability (%)	Reproducibility (%)	LOD (μM)	LOQ (μM)
Glucose	0.05–0.30	0.710	1.32	6.20	2.31	7.74
Fructose	0.05–0.30	0.610	5.03	7.30	2.70	8.15
Galactose	0.05–0.30	0.608	2.55	10.30	2.72	8.20
Lactose	0.05–0.30	0.350	4.97	5.98	4.71	14.28
Maltose	0.05–0.30	0.361	4.91	6.30	4.57	13.85
Mean monosaccharides	0.05–0.30	0.642 ± 0.058	2.96 ± 1.88	7.91 ± 2.10	2.57 ± 0.22	8.03 ± 0.25
Mean disaccharides	0.05–0.30	0.355 ± 0.0078	4.94 ± 0.04	6.14 ± 0.22	4.62 ± 0.12	14.0 ± 0.3

Table 2: Food-sample values used for recovery, dispersion, and dilution-placement calculations.

Food	Direct RS with sensor	Direct RS reference	Total sugar with sensor	Recovery values (%)
Peach juice	44.72 ± 0.40 g/L	46.88 ± 2.10 g/L (TT)	137 ± 16 g/L; reference 140 g/L (LB)	95.4; 97.85
Honey	0.756 ± 0.030 g_{RS}/g_{honey}	0.79 ± 0.004 g_{RS}/g_{honey} (TT)	NT	96.1
Coke	NT	NT	123 ± 17 g/L; reference 120 g/L (LB)	102.5
Diet coke	0 g/L	0 g/L (TT)	0 g/L; reference 0 g/L (LB)	100; 100
Milk	52.6 ± 2.3 g/L	50 g/L (LB)	NT	105.2
Isotonic solution	48.8 ± 3.1 g/L	50 g/L (LB)	NT	97.5
Apple	75.1 ± 0.87 mg_{RS}/g_{apple}	79.82 ± 15.9 mg_{RS}/g_{apple} (Ref)	122 ± 0.72 mg_{RS}/g_{apple}; reference 127.3 ± 29.8 mg_{RS}/g_{apple}	94.1; 96.1

2.4. Cross-Family Calibration-Transfer Bias

In food analysis, a surrogate calibration can be used to analyse the sample when sugar class is unknown. The potential danger of this procedure was measured through calculation of the transfer bias. When sugar class b is estimated with mean slope of the surrogate sugar class a, the concentration is

$$\hat{C}_{b|a} = C_b \frac{\bar{S}_b}{\bar{S}_a}, \tag{6}$$

where $\bar{S}_b$ and $\bar{S}_a$ are the mean sensitivities of the true and surrogate classes. The corresponding percentage error is

$$E_{b|a} = 100 \left(\frac{\bar{S}_b}{\bar{S}_a} - 1 \right). \tag{7}$$

This step converts slope mismatch into concentration bias. It is particularly important for milk, where lactose dominates the reducing-sugar signal, and for fruit or beverage samples where glucose and fructose may dominate. A sensor can show good recovery in a given mode while still being vulnerable to large errors if the wrong class slope is used.

2.5. Apparent Recovery, Conformity, Asymmetry, and Dispersion

For food sample j, apparent recovery was denoted by Rec_j. The apparent-recovery deviation from exact agreement was

$$A_j = Rec_j - 100. \tag{8}$$

A conformity score was retained as

$$G_j = 100 - \left| A_j \right|. \tag{9}$$

This quantity equals 100 when apparent recovery is exactly 100% and decreases symmetrically for over- or under-recovery. Mode-level signed asymmetry was

$$\Omega_m = \frac{\frac{1}{n_m} \sum_{j \in m} A_j}{\frac{1}{n_m} \sum_{j \in m} |A_j|}. \tag{10}$$

A value near zero indicates balanced positive and negative deviations. A negative value indicates a stronger under-recovery tendency, while a positive value indicates a stronger over-recovery tendency. Replicate dispersion was calculated from the mean x_j and standard deviation σ_j:

$$V_j = 100 \frac{\sigma_j}{x_j}. \tag{11}$$

Dispersion was not computed for zero-concentration diet-coke data since the denominator equals zero. The concepts of recovery and dispersion should be viewed together since perfect agreement of the means to 100% does not preclude wide variation among replicates.

2.6. Placing Liquid Sample Dilutions

The dilution factors of liquid samples in g/L were determined from their concentrations. The interior target concentration value was chosen to be $C_T = 0.18$ mM, which is inside the common first interval. The glucose molar equivalent mass 180.156 g/mol was used for the conversion of juice, coke, and isotonic solution samples. Molar mass of lactose 342.30 g/mol was used for milk samples since lactose is the primary reducing sugar in milk samples. The concentration of sample j in g/L, x_j, and its molar mass, M_j, were used to compute the original molar concentration

$$C_j = \frac{x_j}{M_j}. \tag{12}$$

The dilution factor required to place the aliquot at C_T was

$$\Lambda_j = \frac{C_j}{C_T}. \tag{13}$$

The allowable dilution interval for the first calibration window was

$$\Lambda_{j,U} = \frac{C_j}{0.30 \text{ mM}}, \qquad \Lambda_{j,L} = \frac{C_j}{0.05 \text{ mM}}. \tag{14}$$

Any dilution factor between $\Lambda_{j,U}$ and $\Lambda_{j,L}$ places the diluted aliquot within 0.05–0.30 mM. This calculation treats dilution as an analytical design variable rather than a procedural afterthought. The practical aim is to avoid both the lower quantification edge and the upper departure from the first linear window.

3. Results and Discussion

3.1. Sugar-Level Behaviour and Calibration Utility

It is evident that there is a clear distinction among the sugars between monosaccharides and disaccharides. Glucose has the highest sensitivity, 0.710 μA μM^{-1} cm^{-2}, and then come the next two which are nearly equal, namely fructose and galactose with 0.610 and 0.608 μA μM^{-1} cm^{-2}. The two remaining are lactose and maltose having sensitivity values of 0.350 and 0.361 μA μM^{-1} cm^{-2}, respectively. The average monosaccharide sensitivity is 0.6427 μA μM^{-1} cm^{-2}, whereas the average disaccharide sensitivity is 0.3555 μA μM^{-1} cm^{-2}. Therefore, the sensitivity ratio is 1.81. The ratio is very close to the general notion that the monosaccharide response is about twice of the disaccharide response. However, as we will see in the carbon density analysis, the chemical difference between the two groups is more pronounced.

The normalised utility score for the calibration-window ranked glucose the highest. The next two were fructose and galactose with utility scores of 66.8% and 65.4%, respectively. Lactose and maltose comprised the last group with utility scores of 12.7% and 13.9%. The relevant quantities are given in Table 3. The ranking is plausible from an analytical perspective. Glucose is the one that has the highest slope together with lowest LOD and LOQ along with a low repeatability penalty. Fructose exhibits a slightly reduced slope value along with high repeatability penalty whereas Galactose shows a comparable slope value but increased reproducibility penalty. Similarly, lactose and maltose possess close values of sensitivity, detection and quantification; this supports their classification in a paired group.

Table 3: Derived sugar descriptors for class-resolved deployment.

Sugar midrule	Class	S_i (μM-based units)	LOD	LOQ	D_i per carbon	U_i^* (%)
Glucose	Monosaccharide	0.710	2.31	7.74	0.1183	100.0
Fructose	Monosaccharide	0.610	2.70	8.15	0.1017	66.8
Galactose	Monosaccharide	0.608	2.72	8.20	0.1013	65.4
Lactose	Disaccharide	0.350	4.71	14.28	0.0292	12.7
Maltose	Disaccharide	0.361	4.57	13.85	0.0301	13.9

The derived descriptors thus provide an operational ranking. Glucose is the most reliable individual calibrant, although the table also reveals that fructose and galactose are close enough to make up a relevant monosaccharide group. The lactose and maltose calibrants are not weak outliers; rather, they constitute a well-defined lower-slope disaccharide group. Such a classification provides the rationale for class-based interpretation without introducing an additional rule for every individual food sample.

The calibration field in Figure 2 visualises the sugar-level values as a response domain rather than as numerical descriptors. It illustrates that the common first linear interval does not mean the common concentration slope: the three monosaccharides define a high-slope response field, whereas lactose and maltose define a low-slope response field.

The quantification-entry burden offers a useful secondary interpretation. The LOQ values of monosaccharides take only 2.58–2.73% of the 300 μM upper boundary of the common first interval. Disaccharides' LOQ takes 4.62–4.76% of the same upper boundary. Both classes stay analytically feasible, but disaccharides enter the region of measurement later. This distinction is important during sample preparation. A sample rich in lactose should not be diluted to a level close to the lower boundary of the first interval, as the quantification limit of lactose is higher and its slope is lower. On the other hand, a sample rich in glucose or fructose has a safer distance to the lower boundary of the window.

This interpretation indicates that there is no need to abandon the class-based interpretation of electrochemical measurements. The similarity of fructose and galactose allows for the use of the monosaccharide family slope in initial screening of fruit samples. The similarity of lactose and maltose allows for the disaccharide family slope if the disaccharides dominate in a sample. Of course, glucose remains the best individual calibrant for glucose-related accuracy. It means that one should not introduce a family name everywhere to replace sugar-specific calibration. Instead, the presented data reveal where the family approximation is justified and where it is dangerous. This interpretation agrees with the existing electrochemical sensor-array literature proving that family awareness in mixture analysis is beneficial [31,32].

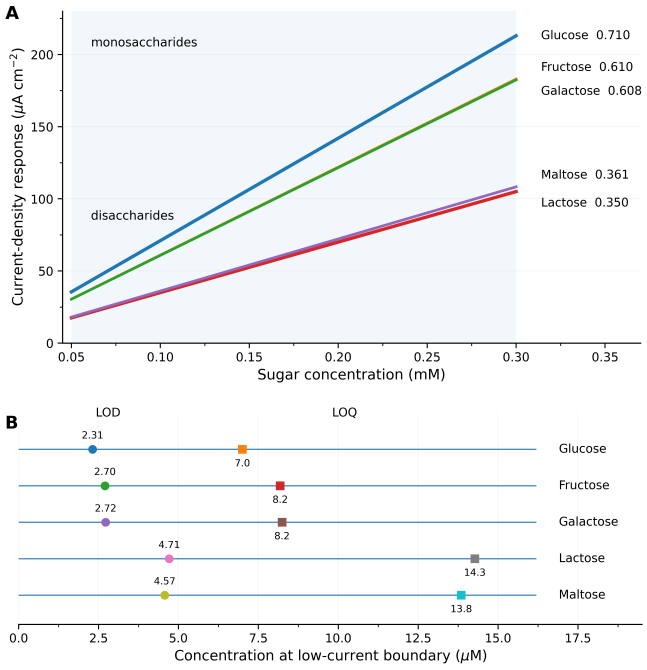


Figure 2: Class-separated calibration field in the first linear interval.

The utility score also allows evaluating the first calibration interval from the quality control perspective. A laboratory analyst usually prefers to conduct measurements in a region away from the lower quantification limit and the upper limit, where the non-linear behaviour starts. Glucose provides the highest numerical safety margin as the LOQ and sensitivity of glucose are the lowest. Fructose and galactose also offer acceptable safety margins, though their utility scores account for the cost of slightly weaker slopes and precision penalty. Lactose and maltose remain measurable, but their low utility scores mean that a disaccharide-rich aliquot should be placed somewhere in the middle of the first interval rather than on its lower edge. This interpretation is invisible by the slope alone. It emerges when the sensitivity, LOD, LOQ, repeatability, reproducibility, and window position are estimated simultaneously.

3.2. Molar-Carbon Response Density and Molecular Interpretation

In fact, this response-per-carbon calculation increased class separation even more. Response density for monosaccharide molecules was 0.1071 μA μM^{-1} cm^{-2}/C. Response density for disaccharides was 0.0296 μA μM^{-1} cm^{-2}/C. Class contrast Ψ_D is thus 3.62. It should be stressed that this value is close to double the raw slope ratio 1.81. Thus, the lower sensitivity of disaccharides to the presence of lactose and maltose cannot be explained just by the fact that these molecules have twice as many carbon atoms as monosaccharides. Rather, the response per each carbon is lower for disaccharides. Graphical representation of this conclusion is shown in Figure 3, where C6 and C12 sugars are

compared with their carbon-normalised current density.

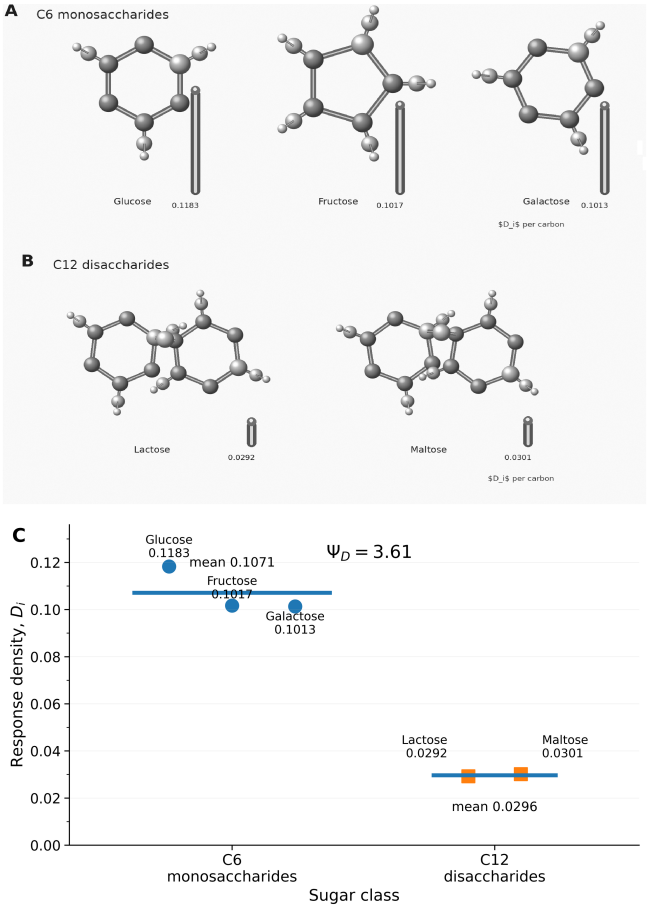


Figure 3: Carbon-normalised response density for C6 and C12 sugars.

This fact has chemical meaning with respect to a NiFe nanowires interface. As a rule, larger sugars have smaller diffusion coefficients than smaller molecules of monosaccharides, and their access to catalytic surface sites is limited due to steric and orientational effects. Lower sensitivity of disaccharides is thus consistent with such processes as slower transport and less favourable interfacial accessibility. Carbon normalisation allows to conclude that disaccharides are not just larger molecules with the same scaled response. They are lower density responders. It means that applying calibration slope for monosaccharides to lactose is not just an approximation but a significant class-related error of the measurement.

The specific surface chemistry of NiFe electrode allows to consider possible mechanism of this effect. In alkaline conditions, nickel and iron centres can form oxidised forms (hydroxide or oxyhydroxide species) that will interact with carbohydrates. The studies of NiFe electrocatalysts emphasise the importance of interaction between nickel and iron in altering redox properties, charge transfer and active site properties [21]. Also, non-enzymatic glucose sensors made from NiFe and nickel-rich materials depend on the access to the surface active sites [20, 21]. Moreover, efficient electron transfer is an important component of the reaction process [22]. If disaccharides reach less productive configuration for oxidation with a lower frequency due to some molecular orientation or diffusion limitation, then lower current per carbon becomes analytically obvious. Although the present study does not include the analysis of specific reaction products, it gives deployment descriptor for the obtained slope difference.

In-class behaviour is also worth considering. Both fructose and galactose show very similar responses, and glucose shows slightly higher response. It means that monosaccharide class is not homogeneous, but the group still shows sufficient homogeneity to allow classification. Lactose and maltose also show very close response, indicating that disaccharides form a stable response group. This information is very valuable in case of the food-quality control, when exact sugar composition may be unknown. For example, fruits beverages can be classified as monosaccharide-rich, while milk is lactose-rich. Using a universal slope for these cases hides all the differences and converts a chemically meaningful response pattern into unnecessary concentration error.

3.3. Cross-Family Concentration Bias

The cross-family bias calculation demonstrates the practical magnitude of class misassignment. If a true disaccharide-rich sample is quantified with the monosaccharide mean slope, the estimated concentration is

$$\hat{C}_{di|mono} = C_{di}\frac{0.3555}{0.6427} = 0.553C_{di}.$$

The concentration would therefore be underestimated by 44.7%. If a true monosaccharide-rich sample is quantified with the disaccharide mean slope, the estimate is

$$\hat{C}_{mono|di} = C_{mono}\frac{0.6427}{0.3555} = 1.808C_{mono}.$$

This error overestimate is 80.8%. See Table 4 for the summary of the errors caused by the family transfer. The errors are much higher than the apparent-recovery deviations in food samples. Such a comparison helps to demonstrate that the main problem with the application of a calibrator is the wrong selection of the calibration curve, rather than deviations from the reference value. Figure 4 demonstrates the results of the error estimation in the form of the bias balance and reveals the asymmetry of underestimation and overestimation.

Table 4: Class-transfer concentration bias obtained from mean calibration slopes.

True sugar class	Surrogate slope used	Estimated/true concentration	Bias (%)
Disaccharide	Monosaccharide	0.553	-44.7
Monosaccharide	Disaccharide	1.808	+80.8

Bias balance allows seeing the direction of error before estimating the values in the table. Quantification of a disaccharide using a monosaccharide slope will compress the measurement while quantification of a monosaccharide using a disaccharide slope will stretch the measurement. The asymmetry is more significant than the small recovery errors since the calculated concentration will change even if the current value is itself reproducible.

Two-row table with bias is very short and important. Its task is not the calculation of yet another parameter of performance but the identification of maximum possible preventable concentration error in calculations. When the slope family is selected properly, recovery rate of food samples is close to reference. When the slope family is chosen improperly, concentration error may exceed one-half of the declared concentration value.

Milk is the best example of this situation. The calibration curve for milk is built according to lactose standard since lactose is the main reducing sugar in this sample. This selection is mandatory. The usage of monosaccharide slope would result in substantial underestimation of the sugar content in milk. The recovery of the direct reducing sugar concentration in milk amounted to 52.6±2.3 g/L, while the concentration declared on label was 50 g/L. So, the recovery of milk concentration was 105.2%. The slight over-recovery of the concentration value compared to the label is relatively unimportant compared to 44.7% underestimation which would occur when the incorrect monosaccharide slope is used. Thus, the milk result is not only a good example of

correct recovery. It is also the proof of the fact that class assignment is a pre-requisite for valid quantification.

The opposite situation occurs when fruit and beverages are studied. The peach juice and isotonic solution are the samples that could be interpreted as the monosaccharide equivalents more realistically. The disaccharide slope usage would exaggerate the concentration of these samples. In a quality control situation, this would cause the wrong conclusion about exceeding declared sugar concentration or about the failure of some technological operation. The bias calculation thus transforms the difference in chemical classes into risk-related measurement statement. Also, it is the proof that the agreement of the results obtained by means of various slope families with standard method is not the reason to use other slope families for any sample. Good agreement in one chemical class does not mean the right to switch into other class.

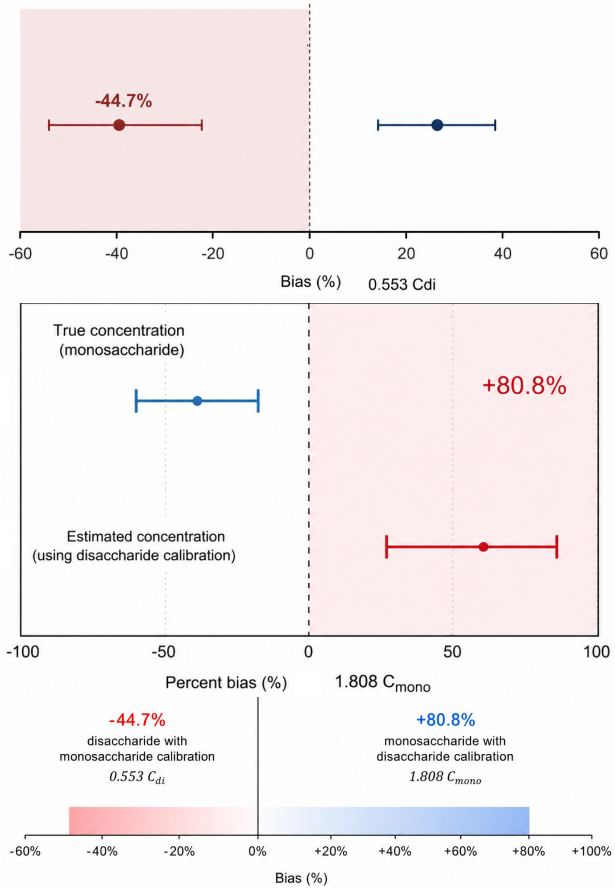


Figure 4: Cross-family concentration bias.

The necessity to select proper slope family for each class of sugars is consistent with the work with non-enzymatic carbohydrate sensors that contain several carbohydrates. Thus, nickel-chromium alloy and nickel nanoflowers were studied as multi-carbohydrate sensors rather than glucose sensors [25, 28]. The renewable copper-based materials were studied as multisugar sensors too [29]. This research shows that this ability is valuable but adds that it should be used in conjunction with the correct concentration conversion. A sensor can detect several types of sugar at once and still require different slopes, pretreatment procedures, or declaration conventions. This is a very important point in food analysis.

3.4. Food-Sample Apparent Recovery and Replicate Dispersion

The recovery of food samples was close to reference values in either mode. This is according to analytical chemistry rules whereby in case of sample preparation, the terminology should distinguish recovery from apparent recovery [33, 34]. Reference uncertainty also constitutes another reason why appropriate use of apparent recovery terminology is necessary [35]. The direct reducing-sugar measurement had a recovery of 98.05% while total-sugar measurement had 99.11%. The mean absolute apparent recovery deviation was 3.68 percentage point for direct reducing-sugar measurement while 2.14 percentage points for total-sugar measurements. The above figures indicate the analytical value of the NiFe nanowires sensor if the proper mode and calibration class are used. It also indicates the possibility for the total sugar pathway to coincide with labels or reference values after conversion of the non-reducing sugar.

The recovery conformity and replicate dispersion profile presented in Table 5 shows recovery conformity and replicate dispersion of each food sample. The direct reducing-sugar mode contains peach juice, honey, diet coke, milk, isotonic solution and apple. The total-sugar mode has peach juice, coke, diet coke and apple. Diet coke showed conformity of 100% in both modes since it was zero in both reference and the sensor. Calculation of dispersion was not possible for diet coke due to its zero mean.

Table 5: Apparent-recovery conformity and replicate-dispersion profile of food samples.

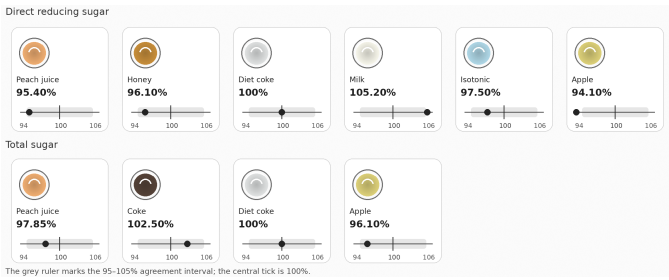
Food sample	Mode	Recovery (%)	G_j	V_j (%)
Peach juice	Direct RS	95.4	95.4	0.89
Honey	Direct RS	96.1	96.1	3.97
Diet coke	Direct RS	100.0	100.0	–
Milk	Direct RS	105.2	94.8	4.37
Isotonic solution	Direct RS	97.5	97.5	6.35
Apple	Direct RS	94.1	94.1	1.16
Peach juice	Total sugar	97.85	97.85	11.68
Coke	Total sugar	102.5	97.5	13.82
Diet coke	Total sugar	100.0	100.0	–
Apple	Total sugar	96.1	96.1	0.59

The recovery table distinguishes between two types of analytical quality. One is agreement with the reference or label value, termed recovery. The other one is the degree to which replicate measurements coincide, called dispersion. Hence, a sample may have satisfactory central recovery yet require careful consideration due to excessive dispersion. Such is the situation in the case of the total-sugar content in beverages. The conversion-dependent preparation procedure increases the chemical completeness of the measurement but worsens replicate dispersion.

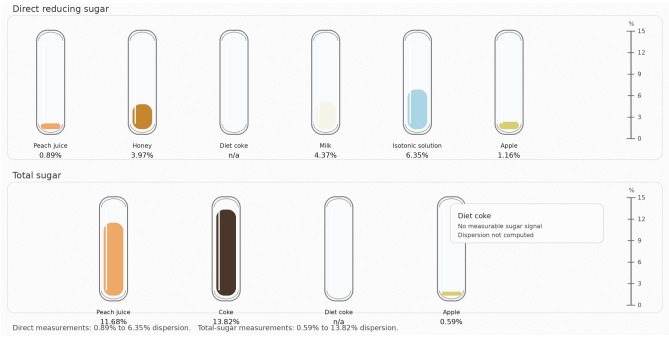
The display of the assay plate in Figure 5 shows the same foods in the recovery-dispersion profile format. The figure clarifies the distinction between central agreement and replicate dispersion and explains why the zero values in the diet-coke samples should be interpreted as negative controls, not as usual coefficient-of-variation values.

The differentiation of the recovery and dispersion makes the interpretation of the total-sugar mode somewhat different. The central recovery was slightly better for total-sugar values, but replicate dispersion was worse. Among the non-zero direct reducing sugar, mean dispersion was 3.35%. In the non-zero total-sugar, the mean dispersion was 8.70%. This does not negate the total-sugar mode. Instead, it means that conversion and dilution procedures cause variability especially in liquid beverages. The total-sugar pathway is chemically inevitable when non-reducing sugars should be quantified. However, it should always go with replicate control. On the contrary, the direct reducing-sugar mode is better for screening when the target is the electroactive

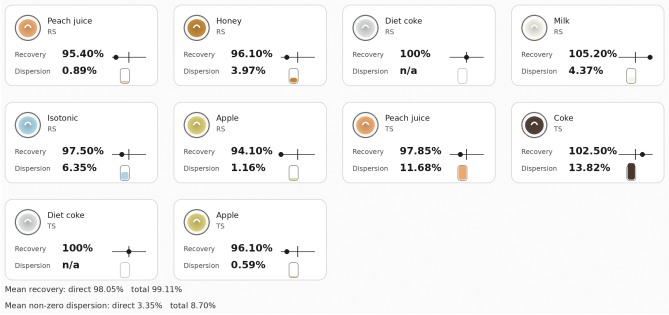
fraction of the sample under selected conditions. Figure 6 illustrates the chemical modes distinction on peach juice and apple when total-sugar treatment causes increase in the measured pool, but not just the repetition of the direct measurement.



(a) Recovery assay plate



(b) Ruplicate dispersion rack

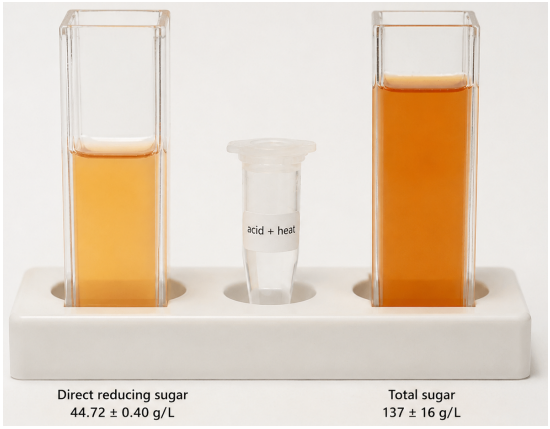


(c) Matric-response tiles

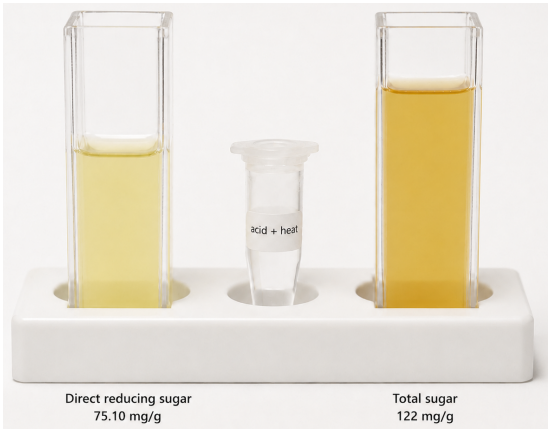
Figure 5: Food-sample recovery and dispersion.

The signed asymmetry gives another piece of information. The signed mean deviation of direct reducing-sugar determinations was −1.95 percentage points and asymmetry was −0.53. In the total-sugar determinations, the signed mean deviation was −0.89 percentage points, and asymmetry was −0.42. Thus, both modes exhibited slight bias to under-recovery but the direction was not strong. Positive deviations in milk and coke partially compensated negative deviations in peach juice, honey, isotonic solution, and apple. Therefore, the asymmetry values should be understood as mild tendency of the mode and not the sign of failure.

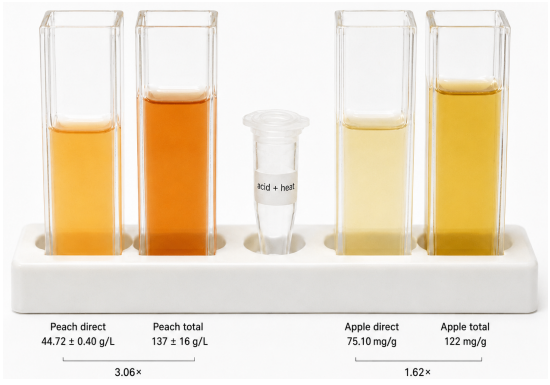
Sample-specific interpretation is more informative than the overall means. The recovery of peach juice in the direct reducing sugar mode was 95.4% and 97.85% in the total-sugar mode. The total-sugar concentration was much higher absolutely: 137 ± 16 g/L against 44.72 ± 0.40 g/L of direct reducing sugars. It is explained by the conversion of non-reducing sugar to the reducing ones. Honey had 96.1% recovery and 3.97% dispersion in the direct mode. It allows the usage of the method but it has the mass-normalised unit and thus, should not be applied to the liquid dilution factor without additional information about the preparation procedure. Milk has slightly high recovery and acceptable dispersion; it should be considered as a lactose-dominant disaccharide sample. The isotonic solution had 97.5% recovery with the highest dispersion in the direct mode: 6.35%. It shows that beverage composition or dilution procedure can influence the repeatability even with good central recovery. Apple had lower recovery in the direct mode (94.1%) and similar but somewhat improved recovery in the total-sugar mode (96.1%), both with low dispersion.



(a) Peach juice



(b) Apple juice



(c) Mode contrast

Figure 6: Direct and total-sugar measurement modes.

The recovery table also explains the reason why negative and unit controls should not be discarded. Diet coke confirmed that the method does not produce the apparent sugar concentration when the assigned value is zero. Honey and apple demonstrate another type of the control – their mass-normalised values cannot be converted to the liquid dilution factors used for beverages. Thus, keeping these unit distinctions prevents the common unit-handling mistake in food-sensor studies: mixing g/L, mg/g and g/g values into a unified concentration processing procedure without explaining the conversion assumptions. That is why the analysis intentionally distinguishes between recovery

interpretation and dilution placement.

The coke sample was presented only in the total-sugar mode and provided 102.5% apparent recovery, but its replicate dispersion was 13.82%, the maximum non-zero value. Such a pattern means that total-sugar measurement may be accurate in the central sense but less consistent in replicate measurements. For industrial quality control, it is important since the decision is made based on the repeated measurements or process trends. Thus, a coke sample with good central recovery but higher dispersion should be processed with enough replicate numbers and consistent pretreatment. Diet coke worked as the negative control. The sensor produced zero values in both direct and total-sugar modes. It confirmed the selectivity to the false positive sugar assignment. Negligible influence of many food-interferents on the NiFe nanowire electrode confirms matrix tolerance [30] in agreement with non-enzymatic sensors literature, which stresses that selectivity should accompany sensitivity [36, 37].

3.5. Liquid-Sample Dilution Placement

The dilution and placement equation represents one of the more practical results obtained during the study. Samples presented as g/L have to be diluted by 10^3–10^4 times for the aliquot to get into the first calibration range from 0.05 to 0.30 mM. The target and allowed dilutions are listed in Table 6. The dilution factor for direct peach juice had to be about 1379 to get down to 0.18 mM. For milk, calculated by lactose content, the target dilution was about 854. Isotonic solution had to be diluted about 1505 times. Total sugar in peach juice had to be diluted about 4225, while total sugar in coke about 3793.

Table 6: Dilution placement for liquid samples expressed in g/L.

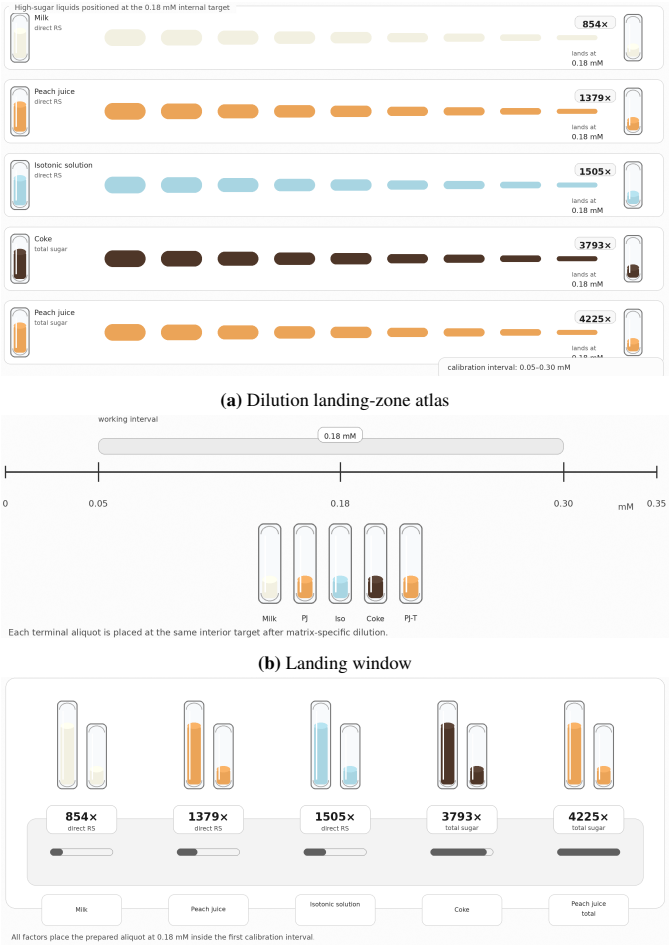
Liquid sample and mode	Target dilution to 0.18 mM	Window-entry dilution to 0.30 mM	Window-exit dilution to 0.05 mM
Peach juice, direct RS	1379	827	4965
Milk, direct RS as lactose	854	512	3073
Isotonic solution, direct RS	1505	903	5418
Peach juice, total sugar	4225	2535	15209
Coke, total sugar	3793	2276	13655

The dilution table is the interface of food composition and electrode calibration in practice. It proves that the technique cannot be applied via direct measurements of undiluted foods but implies that each liquid sample must be transferred into a micromolar range allowing interpretation based on the first calibration interval. This is extremely important for the reproducibility in the laboratory since errors made during sample dilution can significantly grow with thousands of dilution factors.

The atlas of the landing zones (Figure 7) reflects these dilution factors as bands of sample placements. From the figure, one can see that the intention is to move all liquid samples to the same target concentration inside but the food matrix defines the volume of dilution.

These intervals are broad enough for the practical sample preparation. Direct peach juice must be diluted somewhere in the interval of 827 to 4965, and the resulting diluted aliquot will correspond to the first interval. In case of milk, the interval is 512 to 3073. For the isotonic solution, the interval is 903 to 5418. For total-sugar peach juice, the interval is much broader in terms of the absolute dilution factor, 2535 to 15209. In this case, the concentration of total sugar is higher. For total-sugar coke, the interval is 2276 to 13655. The intervals above indicate a two-step procedure. Firstly, apply a sample-type estimate to select an approximate dilution near the target value. Secondly, change the dilution if the measured current is close to the lower or upper boundary of the interval.

This is the clarification of the application of the detection limit as well. The nanowire electrode system has very low micromolar LOD values but in food samples with high content of sugar, the critical question is not if the sensor can detect sugar. The critical question is if the sample is properly diluted in order to have a reliable window of concentration without loosing its class properties. After applying 1000-fold or even 4000-fold dilutions, human errors and equilibration become important. The low LOD is necessary to make the complete analysis possible but it is not the main constraint for these matrices. The main constraint is concentration placement. The micromolar LOD values are enough for most sugar-rich food samples but sensitivity and accuracy in the working range are the key parameters for practical use [30].



(a) Dilution landing-zone atlas

(b) Landing window

(c) Matric-specific dilution factors

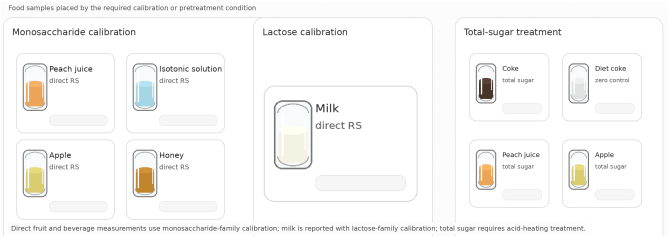
Figure 7: Dilution placement for liquid food samples.

The usage of 0.18 mM as target concentration is conservative. The value is above the LOQ of all five sugars and below the upper boundary of 0.30 mM. The selection does not allow using the lower part of the window which is crowded with disaccharides LOQ values occupying a larger fraction of the usable window. Another laboratory can select 0.15 or 0.20 mM depending on instrument precision and dilution workflow but the principle will be the same. The diluted aliquot must be placed far from the boundaries. It is especially important for total-sugar analysis due to pretreatment increasing dispersion. The calculation provides a conversion of sample concentrations into a reproducible analytical procedure rule.

3.6. Comparisons with Related Non-Enzymatic Sugar-Sensing Strategies

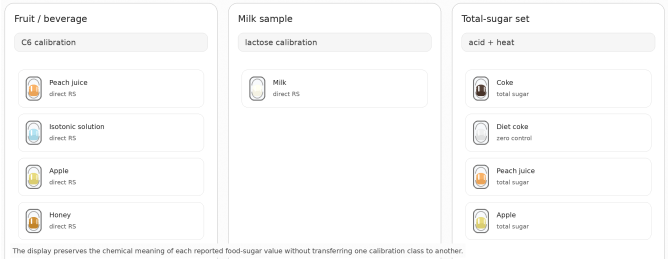
The class-resolved information also serves to compare the NiFe nanowires with related sensors. Several sensors offer reduced detection limits or particular performance for glucose, fructose, or trehalose, but food-quality analysis requires a balanced combination of sensitivity, matrix tolerance, sugar class coverage, and practical

sampling techniques. NiCr electrodes and CuNi thin films represent two transition metal paths to non-enzymatic sugar sensing [25, 26]. NiFe/graphene oxide electrodes and nickel nanoflowers provide other instances [20, 28]. Cu-Ni electrodes and NiCu bimetallic porous structures extend the variety of bimetallic designs [27, 38]. Dendritic Cu-Ni alloy/oxide structures and high entropy alloy structures expand the materials' space [39, 40]. Nanostructured and transition metal non-enzymatic glucose sensors have been reviewed recently, pointing out the persisting issues of catalytic activity and electrode stability [11, 17]. Selectivity and reproducibility are other areas that still deserve attention [12, 18]. Simplicity of fabrication and transferability of the sensor to matrices are essential for practical use [41]. The NiFe nanowire values provide an example of how good a response can be transformed into an operating rule for real food samples.



(a) Class-aware reporting rack

(b) Condition plates



(c) Reporting cards

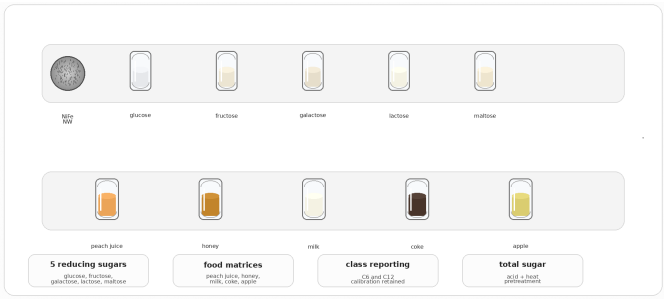
Figure 8: Class-aware food-sugar assignments.

Comparing the sensor with others should involve more than just the technique, linear range, sensitivity, LOD, interfering substances, and names of real food samples used. It should specify the dilution procedure needed to place a real food inside the calibration region and the error to expect when transferring the slope measured for one sugar family to another. The values of NiFe nanowire allow estimating both parameters. Comparison of a food-sugar sensor thus should also involve the type of sugar used for concentration transformation, the dilution range placing representative foods in the calibration range, and the dispersion of replicates of real samples.

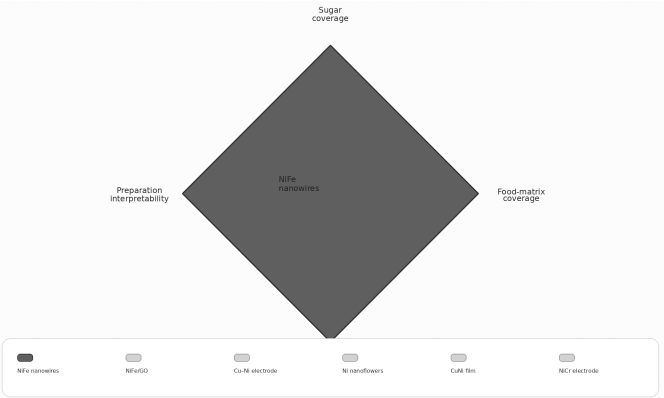
Sensitivity alone is not enough. The sensor based on NiFe nanowires provided a glucose sensitivity of 0.710 μA μM^{-1} cm^{-2} and responded to fructose, galactose, lactose, maltose, and sucrose-derived reducing units. Such broadness is advantageous, since many food samples contain several types of sugars. But the absence of class separation can be misleading in the case of broadness. The cross-family bias values show that selection of an inappropriate slope can lead to the introduction of large errors, which exceed even the apparent recovery deviations found in real samples. The most reliable comparison with other sensors should thus not only include LOD and sensitivity, but also the ability to provide class-specific quantification rules.

The treatment of non-reducing sugar is also important. Untreated sucrose has a very small direct signal, whereas acidification and heating split sucrose into reducing units detectable by the sensor [30]. This is convenient for analytical purposes, since food labelling usually refers to total sugar. Total sugar mode should thus be used in cases where the target is total sugar mentioned on labels or the contribution of sucrose is significant. The price to pay for using total sugar mode is a higher dispersion of replicates in the available data. Measurement of total sugar is thus not a better replacement for direct measurement of reducing sugar. It is another mode with another measurand, another sample preparation protocol, and another precision. Figure 8 represents these differences through reporting territories, while Figure 9 presents the territory of the NiFe nanowire record along with other non-enzymatic sensors.

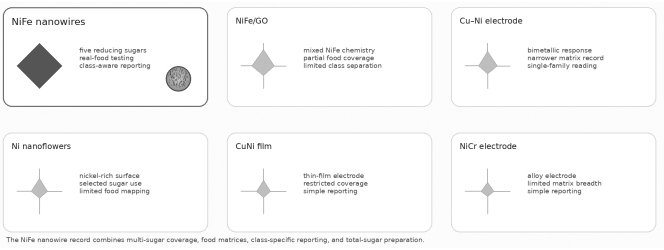
The assignment-rack diagram should be interpreted as a chemical classification table, not yet another step in the laboratory process. Its purpose is to stop analysts from thinking that peach juice, milk, coke, and diet coke all represent the same sugar matrices. A single NiFe nanowire electrode can cover all of these assignments, but each interpretation of the current will depend on the dominant sugar class and total sugar pretreatment.



(a) NiFe deployment record



(b) Deployment breadth compass



(c) Sensor family position cards

Figure 9: Deployment breadth relative to related sensor families.

The deployment breadth compass reveals the position of the NiFe nanowire sensor among its related devices. While some of those sensors make wonderful single-analyte glucose sensors, the present study assesses a different issue: whether the single NiFe nanowire record allows multiple food-sugar modes without losing chemical meaning. So the comparison focuses on deployment breadth and chemical discipline rather than a mere race for the LOD record.

3.7. Operating Rule for Food-Quality Deployment

The combination of results supports a three-part operating rule with the visual assignment shown in Figure 8. First is the choice of the chemical mode of assignment. Fruit-derived reducing-sugar fractions and beverages with glucose-fructose content should be assigned a monosaccharide family calibration or a specific sugar calibration when it is possible and justified. Milk and lactose-containing foods should use disaccharide/lactose calibration. Foods rich in non-reducing sugar must go through total sugar pretreatment before the electrochemical measurement. This step is necessary since the cross-family calibration transfer can cause an error of -44.7% or $+80.8\%$.

Dilution placement is the second step with reference to the landing-zone data in Figure 7. Liquid samples reported in g/L must not be measured without modification. They must be diluted in the direction of an inner point in the first linear interval. The value of 0.18 mM is appropriate because it is above the LOQ of all sugars but below the 0.30 mM limit of the interval. The calculated dilution factors are good starting points: about 854 for milk, 1379 for direct peach juice, 1505 for isotonic solution, 3793 for total-sugar coke, and 4225 for total-sugar peach juice. If the measured current falls outside of the preferred part of the calibration interval, the values can be corrected.

Recovery-dispersion interpretation is the third step corresponding to the food-sample profile of Figure 5. Central agreement near 100% means good recovery, but the dispersion of replicate measurements provides the basis for confidence in decision-making. The direct reducing-sugar measurement had lower dispersion of mean recovery, while total-sugar measurement had stronger central agreement but larger dispersion. Thus, the direct reducing-sugar analysis is better suited for fast screening when the target sugar class is already electroactive. The total-sugar analysis is preferable when the goal is label-relevant total sugar or non-reducing sugar inclusion. Both modes are applicable in the routine quality control, but they should not be lumped together as a single sugar measurement.

The operating rule described above creates a practical deployment framework for the NiFe nanowire measurements. It explains why a highly sensitive sensor still requires chemical judgment, why dilution is an integral part of the technique and not just a preparatory footnote, and why apparent recovery should be assessed with dispersion. The laboratories can validate the rule using the class assignment, dilution window, and recovery-dispersion calculations for their food matrices. In case of NiFe or nickel-alloy electrodes, slope, LOD, and linear range should be complemented with class-transfer bias, dilution window, and recovery-dispersion properties in actual matrices.

4. Conclusion

The research question inquired about the possibility of formulating a chemically meaningful law in which the NiFe nanowire measurements of food-sugar contents would depend on sugar type, dilution position, apparent recovery, and dispersion in the same measurement conditions. The answer is affirmative. The observed calibration descriptors confirm a mode-specific analytical system in which glucose, fructose, and galactose are members of the high-sensitivity monosaccharide class, whereas lactose and maltose belong to the low-sensitivity disaccharide class. The difference between the mean sensitivity values, 0.6427 versus 0.3555 μA μM^{-1} cm^{-2}, is sufficiently pronounced, and the carbon-normalised contrast of 3.62 reveals that disaccharides respond in smaller densities to the interaction with the NiFe nanowires, rather than larger molecules, generating the current scaled up.

The main practical lesson that stems from this study is that the selection of the calibration class represents the dominating preventable source of error. Using the monosaccharide class slope to convert a disaccharide-rich sample leads to 44.7% underestimated concentrations, while the opposite procedure implies 80.8% overestimation. These errors are more significant than the observed discrepancies between apparent recovery values in properly classified food items. Consequently, the numerical evidence answers the question about assignment of the mode to the food result: monosaccharide class of reducing sugar, disaccharide class of reducing sugar, or total sugar following acid hydrolysis.

The food matrix evidence confirms the rule, provided that recovery and dispersion are considered simultaneously. The direct reducing sugar measurements exhibit 98.05% recovery and smaller non-zero dispersion, which makes them appropriate for the fast screening of electroactive sugar fractions. The total sugar measurements confirm the central coincidence with label or reference values via 99.11% recovery, but their non-zero dispersion indicates that the conversion-dependent measurements require increased replicate control. The case of peach juice and apple explains why the classes cannot be mixed up because the treatment of sugar-containing foods with acid increases the amount of the detected sugar.

Dilution position closes the operating answer. High-sugar content liquid foods have to be diluted until the micromolar range of the first calibration interval in order to interpret the NiFe nanowire current. The target dilutions of 854 for milk, 1379 for direct peach juice, 1505 for isotonic solution, 3793 for total-sugar coke, and 4225 for total-sugar peach juice will put the aliquot at about 0.18 mM, far away from the quantification limit and the upper bound of the shared interval. The final answer is thus not that NiFe nanowires detect sugars. The answer is that the NiFe nanowire food-sugar values are reliable enough for quality control if the analyst selects the proper sugar class, dilutes the aliquot into the shared calibration interval, and quotes the recovery with dispersion.

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