

## Literature references

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### Literature references potentiometric titration

- **Cadmium ion biosorption by the thermophilic bacteria *Geobacillus stearothermophilus* and *G. thermocatenulatus***

Adrian Hetzer, Christopher J. Daughney and Hugh W. Morgan

This study reports surface complexation models (SCMs) for quantifying metal ion adsorption by thermophilic microorganisms. In initial cadmium ion toxicity tests, members of the genus *Geobacillus* displayed the highest tolerance to  $\text{CdCl}_2$  (as high as 400 to 3200  $\mu\text{M}$ ). The thermophilic, gram-positive bacteria *Geobacillus stearothermophilus* and *G. thermocatenulatus* were selected for further electrophoretic mobility, potentiometric titration, and  $\text{Cd}^{2+}$  adsorption experiments to characterize  $\text{Cd}^{2+}$  complexation by functional groups within and on the cell wall. Distinct one-site SCMs described the extent of cadmium ion adsorption by both studied *Geobacillus* sp. strains over a range of pH values and metal/bacteria concentration ratios. The results indicate that a functional group with a deprotonation constant pK value of approximately 3.8 accounts for 66% and 80% of all titratable sites for *G. thermocatenulatus* and *G. stearothermophilus*, respectively, and is dominant in  $\text{Cd}^{2+}$  adsorption reactions. The results suggest a different type of functional group may be involved in cadmium biosorption for both thermophilic strains investigated here, compared to previous reports for mesophilic bacteria.

*Applied and Environmental Microbiology* 72 (2006) 4020–4027

- **Industrial implementation of black ripe olive storage under acid conditions**

Antonio de Castro, Pedro García, Concepción Romero, Manuel Brenes and Antonio Garrido

Restrictions on the discharge of chloride in wastewater streams have recently increased, and processors of black ripe olives have to look for alternative storage systems to the traditional brines. Ripe olives of the Hojiblanca variety were stored under aerobic and anaerobic conditions in industrial underground tanks for one year. The aerobic systems assayed with or without NaCl produced a continuous consumption of sugars as they diffused from the fruits to the surrounding liquid. At the same time sugars accumulated in the liquid for months when anaerobic conditions were employed and a high concentration of acetic acid was used. In the end, glucose was consumed with time as well and, in addition to yeasts, acetic acid and lactic acid bacteria grew in the cover solutions. The assessment of olives stored under the different systems and processed as black ripe olives revealed that the traditional aerobic brines gave rise to darker and firmer fruits. However, olive industries must eliminate chlorides from their waste streams. Promising new storage systems were (i) anaerobic preservation of olives in water with an initially high concentration of acetic acid, and (ii) aerobic preservation in water with an initially low concentration of acetic acid. Both systems produced a good quality product, free of microbial spoilage and organoleptic defects.

*Journal of Food Engineering* 80 (2007) 1206–1212

• **Effectiveness of differently designed small-scale constructed wetlands to decrease the acidity of acid mine drainage under field conditions**

P. Kuschk, A. Wiessner, S. Buddhawong, U. Stottmeister and M. Kästner

Constructed wetlands are a near-natural method for the treatment of acid mine drainages (AMD). Because of the different site-specific wastewater qualities and the variability of the used constructed wetlands regarding design, it is difficult to compare their efficiencies on the basis of literature data (often specific removal rates are missing). The AMD treatment efficiencies (pH, acidity) of differently designed planted and unplanted small-scale constructed wetlands (subsurface flow – SSF, surface flow – SF and hydroponic – HP systems with an area of 0.55 m<sup>2</sup> each) were compared under long-term field conditions. The planted SF was found to be most effective, reaching mean acidity removal in the range of 80–90% and most resistant in view of external influences (i.e. heavy rain events). The planted SSF also showed high efficiency (50–90%), but much more sensitivity to rain events. In both systems, the pH increased from 3.3 (mean of the inflows) to above 4.5 in the outflows. The efficiencies of the unplanted SF were insufficient and in the range of the (planted and unplanted) HP, i.e. smaller than 40%. In general, the importance of plants for the success of the neutralization processes could be concluded.

*Engineering in Life Sciences 6 (2006) 394–398*

## Literature references Karl Fischer titration

• **Evaluation of methods for the determination of water in substances with unknown chemical and thermal behaviour**

Dirk Christian Hinz

The water content of six different substances was determined by means of five different methods. The methods used were loss on drying, thermogravimetry (coupled with DSC), a new phosphorous-pentoxide method, direct Karl–Fischer titration and the Karl–Fischer oven. A combination of direct Karl–Fischer titration and the KF-oven method was found to be the optimal technique for water determination of substances of unidentified chemical and thermal behaviour based on detailed considerations about selectivity, type of water binding, sample properties and efficiency.

*Journal of Pharmaceutical and Biomedical Analysis 43 (2007) 779–783*

• **Moisture profile determination in urea prill. I**

Agustin García Barneto and J Ariza Carmona

Using Karl Fischer titration, the moisture profile along the radius in urea prill was determined. Having verified that during dissolution in methanol the urea grains diminish in radius linearly with time, it was possible to calculate that the layer with the highest water content is at a depth of one-third of the radius. The moisture profile is linear, with the central moisture content equal to four times the global moisture content of the grain.

*Journal of the Science of Food and Agriculture 87 (2007) 2217–2221*

• **Multiresponse modelling of the caramelisation reaction**

Mafalda Quintas, Carla Guimarães, João Baylina, Teresa R.S. Brandão and Cristina L.M. Silva

Multiresponse modelling is a powerful tool for studying complex kinetics of reactions occurring in food products. This modelling technique uses information of reactants and products involved, allowing insightful kinetic parameters estimation and helping in clarifying reaction mechanisms. One example of a complex reaction that occurs in food processing is the caramelisation reaction. Caramelisation is the common name for a group of reactions observed when carbohydrates are exposed to high temperatures.

The objective of this work was to apply multiresponse regression in

developing a mathematical mechanistic model that describes sucrose thermal degradation and caramelisation products formation in highly concentrated sucrose solutions, with different water contents and under various temperatures.

Results demonstrated the usefulness of multiresponse modelling in understanding reaction mechanisms in food matrices. A mechanistic model for the caramelisation reaction was proposed, which successfully described the experimental data of concentrated solutions in the 30.03 to 12.20% (w/w) water content range. Furthermore, good predictions of temperature and water content effects were achieved. For extremely low water content systems (3.58% (w/w)), the proposed mechanistic model failed to describe experimental data, indicating different reaction pathways.

*Industrial relevance*

Caramelisation is one reaction occurring during heat treatment in high sugar content food products. Understanding the mechanism of caramelisation reaction and the effect of the environmental conditions on different reaction steps may help in the design of products and processes, in order to prevent or promote such occurrence. This work also uses an advanced modelling technique that can be used in any food system for any reaction occurring during processing.

*Innovative Food Science & Emerging Technologies 8 (2007) 306–315*

## Literature references ion chromatography

• **Chemical analysis of acidic silicon etch solutions II. Determination of HNO<sub>3</sub>, HF, and H<sub>2</sub>SiF<sub>6</sub> by ion chromatography**

Jörg Acker and Antje Henssge

The processing of silicon in microelectronics and photovoltaics involves the isotropic chemical etching using HF–HNO<sub>3</sub> mixtures to clean the surface from contaminations, to remove the saw damage, as well as to polish or to texture the wafer surface. Key element of an effective etch process control is the knowledge of the actual etch bath composition in order to maintain a certain etch rate by replenishment of the consumed acids. The present paper describes a methods for the total analysis of the etch bath constituents HF, HNO<sub>3</sub>, and H<sub>2</sub>SiF<sub>6</sub> by ion chromatography. First step is the measurement of the total fluoride and nitrate content in the analyte. In a second step, H<sub>2</sub>SiF<sub>6</sub> is precipitated as K<sub>2</sub>SiF<sub>6</sub>. After careful filtration of the precipitate, the fluoride concentration in the filtrate is measured and the content of free HF is calculated therefrom. The K<sub>2</sub>SiF<sub>6</sub> is dissolved again and the fluoride content measured and recalculated as H<sub>2</sub>SiF<sub>6</sub>. The results obtained with the presented method are discussed with respect to the results from two other, previously published methods, based on a titration using methanolic cyclohexylamine solution as titrant and based on a method using a fluoride ion selective electrode (F-ISE). An evaluation with respect to the needs for an industrial application is given.

*Talanta 72 (2007) 1540–1545*

• **Polystyrene immobilized ionenes as novel stationary phase for ion chromatography**

Michael P. Raskop, Andreas Grimm and Andreas Seubert

Several aliphatic ionenes (2-6-, 6-6-, 10-6-ionene) have been prepared as ion exchangers for the development of novel high-performance stationary phases for anion chromatography (IC). A macroporous polystyrene/divinylbenzene (PS/DVB) resin with adjusted cation exchange capacity was used as support. Therefore the immobilization of ionenes to polystyrene carriers with remaining positive surface charge became possible for the first time. Strong ion-exchange interactions, resulting in high retention times, between the stationary phase and inorganic as well as organic anionic analytes have been observed. The influence of different ionenes on the retention behaviour during the ion chromatographic separation was investigated. Additionally, partly aromatic and polar ionene backbones were prepared and their retention behaviour as anion exchanger was investigated. The

highest number of theoretical plates obtained was about 90000 per meter. The signal asymmetries were generally lower than obtained for surface functionalized anion exchangers.

*Microchimica Acta* 158 (2007) 85–94

- **Removal of perchlorate from groundwater by the polyelectrolyte-enhanced ultrafiltration process**

Hasina Parvin Huq, Jung-Seok Yang and Ji-Won Yang

The polyelectrolyte enhanced ultrafiltration (PEUF) process for perchlorate removal from groundwater containing common co-contaminants like nitrate and sulfate was investigated. Existing processes such as ion exchange can remove perchlorate but with greater complexity, especially when applied in the presence of high nitrate and sulfate concentration. The effect of cationic polyelectrolyte, poly(diallyldimethyl ammonium chloride) (PDADMAC) concentration and the effect of nitrate and sulfate in a broader concentration range were observed. In the absence of other anions, a lower amount of PDADMAC (0.5–1 mM) can remove more than 90% of perchlorate from the aqueous phase. However, in the presence of 5 mM nitrate, perchlorate removal decreases as the available binding sites of PDADMAC for perchlorate are reduced. In the presence of both 1 mM to 10 mM nitrate and sulfate, 60–80% perchlorate was removed with 30 mM PDADMAC. The removal of sulfate and nitrate in this case was nearly 75% and 65%, respectively. The effect of other parameters such as relative flux, membrane material and pore size were also observed to study the feasibility of PEUF for perchlorate removal. Consequently, PEUF proved to be an effective perchlorate removal process owing to the high removal efficiency and suitability in groundwater remediation containing other contaminants.

*Desalination* 204 (2007) 335–343

## Literature references polarography/voltammetry

- **Ultra trace adsorptive stripping voltammetric determination of atrazine in soil and water using mercury film electrode**

N. Maleki, G. Absalan, A. Safavi and E. Farjami

In situ mercury film electrode produced in the presence of thiocyanate has been shown extremely useful for highly sensitive adsorptive stripping voltammetric measurements of atrazine down to sub- $\mu\text{g L}^{-1}$  level. Operational parameters have been optimized and the stripping voltammetric performance has been investigated using square wave scans. The adsorptive stripping response is linear over the range of 0.5–60  $\mu\text{g L}^{-1}$  atrazine, with a detection limit of 0.024  $\mu\text{g L}^{-1}$ . The method has been applied to the determination of atrazine in soil and water samples.

*Analytica Chimica Acta* 581 (2007) 37–41

- **Inorganic arsenic speciation in water and seawater by anodic stripping voltammetry with a gold microelectrode**

Pascal Salaün, Britta Planer-Friedrich and Constant M.G. van den Berg

The determination of arsenic in sea and freshwater by anodic stripping voltammetry (ASV) was revisited because of problems related to unstable peaks and inconveniently strong acidic conditions used by existing methods. Contrary to previous work it was found, that As(III) can be determined by ASV using a gold microwire electrode at any pH including the neutral pH typical for natural waters. As(V) on the other hand, requires acidification to pH 1, but this is still a much milder condition than used previously. This is the basis of a new method for the chemical speciation of arsenic in natural waters. The limits of detection are 0.2 nM As(III) at pH 8 and 0.3 nM combined arsenic (III + V) at pH 1 with a 30 s deposition time. These limits are lowered by extending the deposition time. The detection step at pH 8 was stripping chronopotentiometry (SC) as this was found to give a lower detection

limit than ASV. Copper is co-determined simultaneously with arsenic. The method was applied successfully to the determination of arsenic as well as copper in samples from the Irish Sea, mineral water and tap water.

*Analytica Chimica Acta* 585 (2007) 312–322

## Literature references stability measurements

- **Physico-chemical characterization of *Moringa concanensis* seeds and seed oil**

Maleeha Manzoor, Farooq Anwar, Tahira Iqbal and M. I. Bhangar

The present work reports the characterization and comparison of *Moringa concanensis* seed oil from Tharparkar (a drought hit area), Pakistan. The hexane-extracted oil content of *M. concanensis* seeds ranged from 37.56 to 40.06% (average 38.82%). Protein, fiber, moisture and ash contents were found to be 30.07, 6.00, 5.88 and 9.00%, respectively. The extracted oil exhibited an iodine value of 67.00; a refractive index (40 °C) of 1.4648; its density (24 °C) was 0.8660  $\text{mg mL}^{-1}$ ; the saponification value (mg of KOH  $\text{g}^{-1}$  of oil) was 179.00; unsaponifiable matter 0.78%; color (1 inch cell) 1.90R + 19.00Y; and acidity (% as oleic acid) 0.34%. Tocopherols ( $\alpha$ ,  $\gamma$ , and  $\delta$ ) in the oil accounted for 72.11, 9.26 and 33.87  $\text{mg kg}^{-1}$ , respectively. Specific extinctions at 232 and 270 nm were 3.17 and 0.65, respectively. The peroxide and p-anisidine values of the oil were found to be 1.75 and 1.84  $\text{meq kg}^{-1}$ , respectively. The induction periods (Rancimat, 20  $\text{L h}^{-1}$ , 120 °C) of the crude oil was 10.81 h and reduced to 8.90 h after degumming. The *M. concanensis* oil was found to contain high levels of oleic acid (up to 68.00%) followed by palmitic, stearic, behenic, and arachidic acids up to levels of 11.04, 3.58, 3.44 and 7.09%, respectively. The results of the present analytical study, compared with those for other *Moringa* species and different vegetable oils, showed *M. concanensis* to be a potentially valuable non-conventional seed crop for high quality oil.

*Journal of the American Oil Chemists' Society* 84 (2007) 413–419

- **The effect of operational parameters of the Rancimat method on the determination of the oxidative stability measures and shelf-life prediction of soybean oil**

Reza Farhoosh

Operational parameters of the Rancimat method, including oil sample size, airflow rate, and temperature, were evaluated to determine their effects on the oxidative stability index (OSI), temperature coefficient,  $Q_{10}$  number, and shelf-life prediction for soybean oil. Operational parameters of the Rancimat method had statistically significant effects ( $P < 0.05$ ) on the OSI. Whenever the oil sample size and airflow rate at a given temperature were such that the air-saturated condition could be established, the OSIs showed no statistically significant differences. As temperature increased, OSIs decreased, while their average coefficient of variation (CV) increased. In general, the conditions where the sample was saturated with air and had a relatively lower CV were an oil sample size of 6 g at all temperatures and airflow rates, then 3-g oil sample size at low temperatures (100 and 110 °C) and low airflow rates (10 and 15  $\text{L h}^{-1}$ ). The temperature coefficient and  $Q_{10}$  number were found to be independent of the oil sample size and airflow rate, and their mean values for soybean oil were calculated to be  $-3.12 \times 10^{-2} \text{ } ^\circ\text{C}^{-1}$  and 2.05, respectively. Oil sample size and airflow rate showed a significant effect on shelf-life prediction for soybean oil. Therefore, choosing the right levels of these operational parameters in the Rancimat method may produce the least possible difference between predictions from long-term storage studies and the OSI test.

*Journal of the American Oil Chemists' Society* 84 (2007) 205–209