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BOOK OF ABSTRACTS

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BUDAPEST UNIVERSITY OF TECHNOLOGY AND ECONOMICS
Department of Organic Chemistry and Technology

Additives and their characterisation for recycling of secondary polymers

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ABSTRACT

This presentation aims to give a complex picture about the polymer additives during the recycling; from the characterisation until the new product. It is obvious, that mainly the polymer type determines the method of the recycling, but in value added recycling also the additives just like fillers, flame retardants and stabilizers play an important role.

In the frame of W2Plastics and Recytech projects the aim was to recover as many pure polymers as possible to recycle them into the production, based on plastic wastes originated from automotive, electrical and electronic, building and construction industries. The end-of-life products are first chopped in a shredder, then separated based on the type of the materials, and finally the shredder light fraction containing plastic waste is classified. Quite pure polymer fractions can be obtained in the case of separation based on densities by floating or magnetic fluid sorting technologies.

One way of the recycling is the value-adding (upgrading), mainly with different additives, which can make the recycling more economic. Before the upgrading step, the comprehensive characterisation of the

secondary plastic density fractions is necessary in order to determine which application they are suitable for. After the determination of the composition, the stage of the degradation and the filler content of each density fractions, the adequate additives can be determined in the view of the requirements of each application.

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Strategied in the pharmaceutical R&D and the Hungarian possibilities

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Methods for real time particle size and shape measurements

H.Pataki

ABSTRACT

The particle size distribution of solids is a crucial parameter in many industry applications, therefore it is particulary important for the effectiveness of the final drug in pharmaceutical industry. The bioavailability of drug can be affected by the liberation of the active pharmaceutical ingredient from preparation in the human body. The dissolution is highly dependent on the crystal size and shape. Besides in the case of active pharmaceutical ingredients (API) and excipients the particle size distribution is significant in terms of production technology of preparation. For instance a solid will be well-tabletting if the crystals are in a suitable size range. If this requirement does not satisfied or crystals have needle-like shape, the preparation of tablets will be often unrealizable. This fact becomes even more significant considering that more than 70% of drugs are sold as tablet.

Crystal size and shape may be controlled by crystallization method. The previous request was that, the crystal size distribution could be tracked in real time on the occasion of crystallization. Why it is so important? On the one side the sample preparation may disturb the „system".

On the other hand we do not know how does a small sample fragment represent the whole crystallization system. In addition during sample preparation may occur unwanted changes in drugs, e.g. in case of high temperature samples might be „recrystallized".
Nowadays there are several in-situ monitoring tools for different particle properties determination. The focused beam reflectance measurement (FBRM) technique introduced by Lasentec and in-situ microscopy, e.g. applied using a process video and measurement (PVM) microscope, are in-situ methods, often used to monitor the evolution of particle populations. Ultrasonic spectroscopy is also applicable to determine the crystal size distribution in real-time during crystallization processes.

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Introduction to multivariate data analysis (chemometrics)

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ABSTRACT

Chemometrics is a field of mathematics dealing with large datasets and databases. A dataset of countries and their economic indicators (wage rate, GNP, level of inflation, amount of debt) is used as an example in the presentation. The Raman spectra, collected in our research group, form a similar type of dataset.

An overview of the different fields are given within chemometrics. Clustering and classification methods can be used to form groups of the objects (these object can be the countries in the example or can be Raman spectra in spectroscopy). It is shown in the example dataset that the economic indicators are highly correlated with each other: the number of true underlying variables can be determined by factor analysis. In addition, regression methods are also shown in an example where we want to predict the wage rate in a country using the other indicators (GNP, debt and inflation). Examples of the above mentioned methods are given for Raman measurements.

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On-line Raman spektrométerrel összekapcsolt, számítógéppel szabályozott kristályosító berendezés fejlesztése

P. Bárány

ABSTRACT

A kristályosítás mostanában az egyik legfontosabb gyógyszeripari eljárássá válik. Ennek oka, hogy a korábbi szemléletmódtól eltérően már nemcsak egyszerű végtermék tisztítási lépésként, hanem olyan technológiai lépésként ismeretes, ahol az alkalmazott kristályosítási paraméterek meghatározzák a kristályos anyag számos tulajdonságát. A folyamat körülményeinek változtatásával befolyásolható a kristályszerkezet, szemcseméret, szemcseméret eloszlás (CSD: crystal size distribution), ill. kristály habitus. Egy anyag különböző kristályszerkezetű módosulatait, azaz polimorfjait eltérő a fizikai és kémiai stabilitás, oldhatóság és biohasznosulás jellemzi, így az egyes formák szabadalmaztatása a gyógyszerfejlesztés korai szakaszában kiemelkedő fontosságú. A kristályok alakja, mérete, szemcseméreteloszlása, folyási tulajdonsága, préselhetősége pedig többnyire a gyógyszerformulálás egyes lépéseiben meghatározóak, illetve a hatóanyag felszívódásában is szerepet játszhatnak a gyógyszerformából történő kioldódásuk révén. Mindezek ismeretében

nem meglepő az elmúlt években a gyógyszeriparban megjelenő törekvés, mely a kristályosítások szabályozhatóságát tűzte ki célul.
A kristályosodás speciális körülményeinek biztosítására olyan számítógéppel irányított laboratóriumi rendszer fejlesztését tűztük ki célul, amely a szakaszos és félfolytonos kémiai folyamatok tanulmányozásán túlmenően alkalmas a szokásos gyógyszeripari reaktorok (kristályosítók) modellezésére is. A keverés fordulatszáma, a hőmérsékletet, a pH és a reagensek adagolása egyaránt szabályozható. Az exoterm és endoterm entalpiaváltozások követhetők és meghatározhatók, így optimalizálhatjuk a folyamatot, ily módon növelhetjük a termelést és csökkenthetjük a reakcióidőt. A kristályosítás nyomon követésére valós idejű Raman spektrometriás nyomon követését végzünk. A Raman spektrumok változása alapján következtetni lehet kémiai átalakulások mellett a kristály-módosulatváltozásokra is.

A folyamatirányított rendszer kifejlesztésénél felhasználjuk a Tanszéken korábban kialakított reaktorkaloriméter fejlesztésénél szerzett tapasztalatokat, valamint egy professzionális laboratóriumi folyamatirányító rendszer (Labmax) és egy in-situ FT-IR (ReactIR) felhasználása során szerzett ismereteket. A rendszer kifejlesztésében ipari szakemberek is támogatást nyújtanak (Richter – Vaczulin Zoltán, Yokogawa – Szelmann Szilárd, Kocsis András).

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Multilayer flame retarded composites from recycled automotive shredder plastic waste

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ABSTRACT

In connection with the increasing consumption of polymeric materials in automotive manufacturing, the amount of automotive shredder plastic waste increases simultaneously. This situation creates a large burden on the natural environment, because increasing area is required for waste disposal, or during their burning toxic gases are forming. Managing the plastic waste is one of the challenges faced by today's world.
Secondary plastics have a low market value because of their low purity and therefore their recycling is not economic yet. The transformation of waste to materials for engineering purposes (as modified composites or blends) can be the way which provides a quality increase instead of quality deterioration (degradation, down-cycling) experienced in recycling.
Upcycling possibilities of the density fractions of separated automotive shredder polymer waste (received in the frame of the W2Plastics EU7 project) were studied. In the field of transportation mechanical and flame retardant properties play an important role in the material development, but with application of flame retardant additives, the

mechanical properties considerably deteriorate. In order to fulfil these antagonistic requirements sandwich structured polymer composites consisting of basalt or glass fibre reinforced core and flame retardant phosphorus-containing intumescent shell including polyurethane and mixed polyolefin waste were developed. The results of different layered composites were evaluated using standard methods for mechanical and flame retardancy investigation.

The application of the flame retarded shell material successfully decreased the peak of HRR of the sandwich composites and increased the LOI from 19 of the core material to 26 in case of the recycled sandwich composite by retaining the same UL-94 level. The application of the recycled shredder automotive waste instead of virgin materials did not cause considerable deterioration in terms of the fire retardancy in case of the flame retarded shell and the sandwich composite, moreover it was advantageous due to its high filler content. Based on the results of mechanical tests it can be stated that in case of the recycled materials the properties decreased by 30% compared to the reference material, but the basalt and glass fibre as reinforcement has effectively increased the mechanical properties of the recycled matrix composite, consequently the mechanical properties of the prepared sandwich composites containing recycled materials are still proper for engineering applications.

On the basis of our work, the effective and economic up-cycling of automotive shredder plastic wastes is possible. The simultaneous amelioration of mechanical properties and fire resistance of automotive plastic waste was accomplished by preparing fiber reinforced sandwich composites upgraded with flame retardancy. The developed composites can be suitable for several further application possibilities; consequently they can be also marketable.

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Erősen átfedő spektrumok megkülönböztetése kemometriai módszerekkel

A. Farkas

ABSTRACT

Keverék minták Raman-spektrumai egyszerűen modellezhetők, amennyiben rendelkezésre állnak a keveréket alkotó komponensek tiszta spektrumai. Amennyiben ezek nem állnak rendelkezésre, a Raman-térképből kell ezeket megbecsülni kemometriai módszerekkel (főkomponens-analízis - PCA, önmodellező keverékelemzés - SMMA, többváltozós görbefelbontás - MCR-ALS). Mindegyik módszer jó eredményt ad, ha a keverék kémiailag különböző anyagokból áll, melyeknek a spektrumai nagy mértékben különböznek egymástól. Munkám során azt vizsgáltam, hogy igen nagy hasonlóságú spektrumok (pl. különböző gyógyszerpolimorfok spektrumai) is elválaszthatók egymástól.

Két polimorfról (carvedilol A és B) felvett spektrumok alapján a polimorfok 50-50%-os fizikai keverékének kémiai térképét modelleztem. Így a térkép egyes pontjain megbecsülhető váltak a komponensek koncentrációi.

A két polimorf spektrumának ismeretében úgymond mesterségesen (Matlab segítségével) is előállítottam egy térképet. Ebből a térképből

megkíséreltem az előzőleg felsorolt kemometriai módszerekkel visszakapni a két komponens Raman-spektrumát. A módszerek hatékonyságát, alkalmazhatóságát különböző zajszintek mellett is felmértem.
Az erősen átfedő spetrumok esetén a PCA nem volt alkalmazható. A SMMA 30%-os zajszintig adott megfelelő spektrumokat, míg a leghatékonyabb MCR-ALS még a 100%-os zajjal terhelt térképspektrumokból is elfogadhatóan adta vissza a két polimorf spektrumát.

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Multivariate methods for quantitative analysis

I. Farkas

ABSTRACT

The use of vibrational spectrometry in quantitative analysis is widespreadly increasing nowadays. These methods make the on-line measuring possible, and provide better estimations of chemical composition. However, these measurements are indirect and rely on the ability to develop a model that relates the set of measured variables to the chemical compostition of the system. These models are often used in spectrometric applications because of the lagre amount of information available in spectrometric data.

The Classical Least Squares (CLS) model is based on the weighted sum of linearly independent signals. In Raman spectrometry the CLS model assumes that measured spectra are the sum of pure component spectra weighted by the concentration of the analytes. The main disadvantege of CLS is that all of the spectrally active components must be known. These estimates must include any minor componentes that may not be of interest themselves but may contribute to the measured signal. Further disadvantage, if the concentrations of the analytes in the calibraton spectra is not known, the predictions will suffer considerably.

The Partial Least Squares (PLS) regression model is a factor analysis-based model. PLS attempts to find factors which both capture variance and achieve correlation. PLS attempts to create new variables that maximize covariance with the attribute to be predicted (in this case the concentration).

Spray dried samples with different compositions were prepared and were investigated with micro-Raman spectrometry. Univariate (based on band area), CLS and PLS regression models were tested to see, which provides the most accurate determination of caffeine content. Genetic algorithms (GA) were applied to further improve the PLS results. The results were compared to Differential Scanning Calorimetry (DSC) analyses.

In conclusion, all the above mentioned regression methods are able to predict the concentration of the analytes. Best results were achieved using PLS regression after filtering the non-useful variables with GA.

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Applications of The Extrusion Techniques

T. Horváthová

ABSTRACT

The melt extrusion technology has been being already used successfully for food engineering and processing of polymers, respectively for decades. Mainly the extrusion was applied only for wet granulation and spheronization, subsequently in the industrial medicine production but recently even growing interest is shown by the pharmaceutical industries for the wide range of the application of the continuous melt extrusion technology. This technique can be efficient for enhancing the dissolution rates for poorly water soluble drugs, modifying the drug release furthermore processing drug delivery systems.

The preparation of the molecular dispersion of an active ingredient in a hydrophilic polymer is a powerful way for decomposing the crystalline structure of the drug and improving its solubility, consequently.

My student academic work aimed the preparation of solid solution of the poor water soluble spironolactone via twin-screw extrusion [1] process. The binary adducts of the drug and hydroxypropyl-β-cyclodextrin were prepared by solvent co-evaporation. DSC, Raman and FTIR studies indicate the absence of crystallinity of drug due to the interaction.

The forming of the interaction in extrudates was investigated by Raman-, FTIR-spectrometry and XRPD. The influence of the screw rate on the inclusion complex was evaluated and an optimal rotation speed was found at which the fine dispersion of spironolactone can be obtained in the matrix moreover the residence time is quite long for the interaction between spironolactone and hydroxypropyl-β-cyclodextrin.

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Amorphization of spironolactone by lipid extrusion

T. Jordán

ABSTRACT

The solubility behaviour of drugs remains one of the most challenging aspects in formulation development. With the advent of combinatorial chemistry and high throughput screening, the number of poorly water soluble compounds has dramatically increased.

Melt extrusion may be applied to disperse drugs in a given matrix down to the molecular level, e.g. to form a true solution. It is the convenience of the technology that gives new hope to the glass or solid solution approach as a delivery system for poorly soluble drugs.

The aim of my work was to dissolve the poor water soluble spironolactone in a sufficient tenside and prepare its solid dispersion [1] in a hydrophilic polymer matrix simoltanously through the course of twin-screw extrusion [2].

Melted Gelucire 50/13 and Precirol were applied to dissolve the spironolactone, then the process temperature furthermore the extrudable ratio of Gelucire 50/13 and Aerosil 200 was determined by viscosity studies. Maize starch plasticised by sorbitol was applied as hydrophilic outer phase for forming solid dispersions via twin-screw extrusion. Suspension like extrudates can be carried out by adding Aerosil 200

which adsorbs the tenside drops including dissolved spironolactone. If the composition absenced the nanofiller the temperature had to be reduced in order to avoid the phase separation of the melted Gelucire and the plasticized polymer.
The formed molecular interaction in extrudates was characterised by Raman-, Fourier transform infrared (FT-IR) spectrometry and differential scanning calorimetry (DSC).

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Characterisation of polymer wastes originating from automotive industry

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ABSTRACT

Shreddered plastic waste originating from automotive industry was received in the frame of the W2Plastics - EU7 project (Magnetic Sorting and Ultrasound Sensor Technologies for Production of High Purity Secondary Polyolefins from Waste). The aim of this work was to recover as many of quite high purity secondary plastics as possible to recycle them into the industrial production focusing mainly on polyolefins. This study presents the results we had obtained by analysis with FTIR Spectrometer, DSC, LP-FTIR and Raman Spectrometer, using polyolefin containing samples originated from auto recycling. The samples were separated using the principle based on the difference of density by flotation in ethanol solution in the range of 0.88-1.4 g/cm³. The purpose of this analysis was to determinate the type and the composition of polymers and fillers in samples, in order to make it capable to reuse depending on their properties. According to FTIR and

Raman results separation density limits were determined to obtain reasonably homogeneous fractions to recover polypropylene (0.88-0.93 g/cm^3), polyethylene (0.93-0.97 g/cm^3). The presentation contains also the estimation of the price of polymers as a result of a process of recycling and separation.

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Restabilization of secondary polyolefins

Kinga Madi

ABSTRACT

Four main possibilities are known for plastic waste treatment: deposition, burning with or without energy recovery, and chemical or mechanical recycling. In the last decade laws and regulations appointed to reduce the amount of plastic waste for deposition [1].
In the course of my work I have participated in W2Plastics (EU7) and Recytech (Hungarian) recycling projects aiming the recovery of pure polyolefin waste fractions originated from automotive, electronic and building industries. Main target of my research is the value-added recycling of these industrial polymer wastes.
The purpose is the development of the recyclates for several applications by upgrading with fire retardancy, stabilization and reinforcement. The improvement of properties can be verified by mechanical, rheological and combustibility measurements.
In the present work stabilizers were tested in different concentrations on reference polypropylene and also on plastic wastes containing mainly polypropylene [2]. With restabilization the aim was to protect the polymer from degradation during processing and reuse. Although several mechanisms of polymer degradation are known, high temperature and the presence of oxygen favour the thermooxidative degradation during the processing.

Degradation has a great influence on the properties of plastics. Molecular weight will change, and therefore also the melt flow viscosity, mechanical properties will decline, and it will lose its original colour [3]. Number of carboxyl groups will increase as function of time, which can be detected by FTIR.
We performed model measurements with virgin materials and also with waste. The effects of the stabilizers were tested. Some samples were aged in an aging chamber at 95 °C others were homogenized seven times with a double-screw extruder. After aging or multi-extrusion oxidation induction time (OIT), melt flow index (MFI) and yellowness index (YI) were measured. In order to examine the change of mechanical properties; fracture tests, bending tests and tensile tests were also performed.
In the future this work may be of assistance in finding an appropriate solution for plastic waste recycling.

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Application of electrostatic spinning in formulation of nanostructured drug delivery system

K. Nyúl

ABSTRACT

Nowadays one of the largest challenges in the field of pharmaceutical technology is the enhancement of drug release from orally taken solid dosage forms as most of the recently developed APIs have poor water solubility [i]. In the period of nanotechnology there are new possibilities to improve dissolution of these already developed and candidate drug molecules. Electrospinning is a relatively new technique in the field of pharmaceutical technology and it was mainly applied to achieve sustained drug release [ii].
In this work DonepezilHCl with good water solubility and Carvedilol with poor water solubility were used as model drugs to investigate the capabilities of electrospun materials to improve the dissolution rate.
Poly(vinyl-alcohol) (PVA) and poly(vinyl-pyrrolidone) (PVP) were dissolved in water or in methanol with the model drugs and these solutions were electrospun onto carriers. Morphology of the samples was investigated by AFM, SEM and optical microscope. Chemical properties and crystal morphology were characterized by Raman-microscopy, FT-IR, DSC and XRPD. In vitro drug release was followed by UV-VIS spectrometry.

The diameters of obtained fibres were 100-200nm in the case of PVA, while the drug loaded PVP fibres were somewhat thicker 500-900nm. The electrospun samples were dissolved by water immediately and drug release time was no more than half minute. Dissolution time of the other "traditional" films was more than half an hour, which is much longer than that of electrospun mats. According to XRPD, Raman-microscopy, FT-IR and DSC investigation the APIs were in amorphous state in the polymer matrix.
This work shows that this technique is able to fabricate mats with ultra-fast drug release from poorly water soluble drug (Carvedilol) and the production in industrial scale is available because it was carried out in the recent years in the textile industry.

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Crystallization of Carvedilol: influence of polyvinyl-pyrrolidone

K. Palásti

ABSTRACT

Crystallization in a presence of additives like polimers is a relatively less explored area, but it is important in polymorphic screening of a drug during its developmental stage. The presence of small amounts of additives in a crystallization medium dramatically can change the crystal shape and size, rarely polymorphism.

In this study the influence of polyvinyl-pyrrolidone on the Carvedilol's crystallization was investigated. The effect of this polymer additive was examined in antisolvent and cooling crystallizations. All experiments was carried out in ethanol solvent, occasionally water was used as an antisolvent. In the presence of 1% polymer nucleation was already delayed in colling crystallization experiments, the solid active ingredient appeared one day after stored in the refrigerator. The mechanism of the nucleation retardation by the polymers is explained in terms of association of Carvedilol with polyvinyl-pyrrolidone through hydrogen bonding.

Habit modification was observed in antisolvent crystallization, when the dissolution of the API was not complete. At this time rose-like crystal formations grew from the solution, due to the retardation of the crystal face growing in certain directions. This latter effect may be attributed to polymer.

In the course of crystallization in ethyl acetate with low drug concentration a new solvate/ansolvate form was observed, which is transformed into a more stable, Form III. polymorph after three days.

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Development of multilayer flame retarded epoxy resin composites

B. Szolnoki

ABSTRACT

The applicability of phosphorus-containing reactive amine, which can be applied in epoxy resins both as hardener and as flame retardant, was compared in an aliphatic and an aromatic epoxy resin system. In order to fulfil the strong requirements on mechanical properties of the aircraft and aerospace applications, where they are mostly supposed to be applied, carbon fibre reinforced composites were prepared. The flame retardant performance was characterized by relevant tests and mass loss type cone calorimeter. Besides the flame retardancy, the tensile and bending characteristics and interlaminar shear strength were evaluated. The intumescence-hindering effect of the fibre reinforcement was overcome by forming a multilayer composite, consisting of reference composite core and intumescent epoxy resin coating layer, which resulted in simultaneous improvement of flame retardancy and mechanical properties of epoxy resins.

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Embedding of Lactobacillus acidophilus bacteria into PVA nanofibers

I. Wagner

ABSTRACT

Probiotics are even more and more widely used in health care which has several causes. On one hand probiotics have several positive effects, like the interaction with the immune system, anti-cancer potential, and potential as a biotherapeutic agent in cases of vaginitis, inflammatory bowel disease and irritable bowel syndrome. On the other hand patient compliance is much better in the case of biomedicines compared to synthetic drugs due to the gaining public acceptance of "bioproducts". Moreover biodrugs has less toxicological risk from the point of view of the developer and as regard the consumer they have fewer side effects.
However it is necessary to use adequate dosage forms of biodrugs to provide appropriate effect. In case of classically used probiotics the controlled dosage means a serious challenge for pharmaceutical technologists what can be solved using solid medicines. In general the main critical step during the production of solid probiotics is drying. It has to be performed under mild circumstances because of the nature of these materials. A generally used method is the lyophilisation that is not only energy intensive but also time consuming.

In this study electrostatic spinning technology (ES, electrospinning) was investigated as a competitive and gentle drying method for formulation and preservation of lactobacillus acidophilus (LBA) which is a prominent member of probiotics. By this method even the drying of products containing bacteria is possible. In our experiments we embedded LBA into PVA nanofibers using electrospinning. The reference product was Gynoflor® which contains 50 mg freeze-dried LBA with ten million CFU (colony forming unit). Our aim was to reach this number of cells with electrospinning. LBA was subcultured on oblique MRS agar. Electrospinning was carried out by making a suspension of bacteria in the polymer solution. Morphology of the electrospun materials were investigated by optical microscope and scanning electron microscope (SEM). We also studied the effect of the process on the bacteria so they were enumerated after the electrospinning and stability were studied at 25 °C, 7 °C and -20 °C. The result of the enumeration showed that more than the two-thirds of the bacteria survived the method of embedding.

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