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A noninvasive NMR and MRI method to analyze rehydration process for dried sea cucumber

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Sea cucumbers possess high-value and bioactive components that have been used for human food and pharmaceuticals in treating a wide number of ailments. Most of the sea cucumber products in the market are dehydrated due to the autolysis problem. Rehydration of the dried products is necessary for obtaining the best rehydrated sea cucumbers with the most water content. In this study, a rapid and non-invasive NMR and MRI method was introduced to analyze rehydration process of dried sea cucumber. The spin-spin relaxation time (T_2) weighted NMR signal, obtained by a CPMG pulse sequence and processed by chemometric method, was used to identify lightly dried and salted dried sea cucumber. The water uptake and distribution during rehydration process was monitored by NMR ¹H T_2 . The structure change was analyzed by MRI with T_1 and T_2 weighted imaging. The results indicated that the proper presoaking and rehydration time was 24 and 96 h, respectively, for the lightly dried sea cucumber. Good linear correlation during the rehydrated process was observed between the NMR parameters and texture profile analysis parameters including the hardness, chewiness and rehydration ratio of lightly dried sea cucumbers. The NMR and MRI method has the potential to noninvasively analyze the rehydration process of dried sea cucumber.

1. Introduction

Sea cucumber is one of a kind of the marine animals, belonging to the class Holothuroidea, usually found in the benthic areas across the world.¹ It has long been used for human food and pharmaceuticals because of its high-value and bioactive components, particularly in some parts of Asia.² In the market, more than 80 % of the fresh sea cucumbers, harvested all over the world, are processed to produce a dehydrated product due to the autolysis problem.³ Although the nutritional value of dried sea cucumber is not good than that of the fresh sea cucumber, the dry products can be easily transported at normal temperatures and stored for a long period of time. Briefly, the dehydrated sea cucumber products include the lightly dried and salted dried sea cucumber. It is very important to analyze the rehydration process of dried sea cucumber because this process can significantly affect its physical and biochemical status leading to change the color, texture and taste and causing loss of nutrients. Therefore, in order to reduce the rehydration time and obtain an appropriate rehydration method, it is necessary to develop a fast, non-invasive method to analyze the rehydration process of dried sea cucumber.

Rehydration of dehydrated food is a complex phenomenon, which is affected by numerous factors including medium characteristics, presoaking time, number of rehydration times, size of food sample, drying method of food sample, etc.⁴ It is therefore expected that the water status in dehydrated food is monitored during the rehydrating process. However, as previously indicated, non-invasive and fast analytical method is lacking to gain the detail information of the rehydration process.⁵ Duan *et al.*⁶ studied the rehydration ratio and texture characteristics of dried sea cucumber using physicochemical methods. This method is tedious, expensive, time-consuming, and require skilled personnel. For this reason, the development of a rapid, non-destructive, low-cost control, analytical method would be a priority for the industry.

Low field nuclear magnetic resonance (NMR) has become a powerful tool in monitoring the food processing because of its non-invasive characteristics, high reproducibility, and sensitivity.⁷ In general, NMR is based on the measurement of resonant radio frequency absorption by non-zero nuclear spins (protons have the spin I=1/2) in the presence of an external static magnetic field.⁸ The analytical method of NMR reflects distribution of water pools with different relaxation time in food.⁹ The features of proton relaxation are characterized by spin-lattice relaxation time (T_1) and spin-spin relaxation time (T_2) .¹⁰ NMR has been used to identify food samples, such as honey,¹¹ beef,¹² and sweet corn,¹³ based on hydrogen proton difference. On the other hand, magnetic resonance imaging (MRI), another type of NMR technology, has been clinically used in detecting structural abnormalities of the body,14, 15 which has also been used to explore the food analysis and food processing.^{13, 16} It is considered to be an accurate and nondestructive method for visualizing the internal food structure.¹⁷ There has also been a significant effort to use both NMR and MRI for food analysis and food processing. For example, water uptake, mobility and distribution in biological macromolecules systems have been conducted using NMR or MRI.¹⁸⁻²⁰ Liu et al.²¹ used both NMR and MRI to study the gelling properties of egg white gel. Zhang *et al.*¹⁰ monitored the water uptake at macroscopic and microscopic levels during cooking of navy beans by using MRI and NMR relaxometry. The combination of NMR and MRI in these food processing has prompted us to think that NMR and MRI might be useful for identifying the dried sea cucumber and analyzing their rehydration process. This method may provide more information, such as water uptake and distribution, internal structure change of the sea cucumber samples at the same time, which can't be achieved with any other method.

In this study, a rapid and non-invasive NMR and MRI method was introduced to analyze rehydration process of the dried sea cucumbers. The T_2 weighted NMR signal obtained by a CPMG pulse sequence and processed by principal component analysis (PCA) was used to identify the lightly dried and salted dried sea cucumbers. The water uptake, distribution and structure change of the lightly dried sea cucumbers during presoaking and rehydration process were analyzed by the NMR and MRI. The relationships between the individual T_2 relaxation parameters and rehydration ratio, texture characteristics of dried sea cucumber during rehydration processing were investigated with NMR. The method by using both NMR and MRI for analysis of dried sea cucumber rehydration may have great potential for rapid and non-invasive analysis of other dried food processing.

2. Material and methods

2.1 Sea cucumber samples

Dried sea cucumber samples were purchased in local market and stored in the sealed environment for the experiment. The sample consisted of lightly dried sea cucumbers and salted dried sea cucumbers. The weight of each dried sea cucumber sample is about $2.68 (\pm 0.27)$ g.

2.2 Rehydration process

In order to investigate the time effects, the lightly dried sea cucumbers were presoaked with deionized water for 4, 8, 12, 16, 20, 24 and 28 h, respectively, at room temperature. NMR T_2 relaxation time spectra and MRI pictures of the presoaked sea cucumbers were measured at different time points. Then, the sea cucumbers were boiled for 30 minutes and equilibrated to room temperature, followed by rehydration with deionized water for 24, 48, 72, 96, 120 and 144 h, respectively, at 4 °C. NMR T_2 relaxation time spectra and MRI pictures of rehydration process at different time points were measured to monitor the water distribution and structure change of sea cucumbers.

Water absorption of sea cucumbers was determined after every 24 h of the rehydration. The rehydrated sea cucumbers were blotted with a paper to remove free water on their surface. Rehydration ratio was expressed as a percentage of water absorption and calculated by the following equation. Percentage rehydration ratio = $(m_g - m_0)/m_0$, where m_0 and m_g are the weights of before and after rehydration, respectively.

2.3 NMR analysis

Transverse relaxation T_2 measurement was performed on MiniMR-Rat (Niumag Electric Corporation, Shanghai, China)

equipped with a 0.5 T permanent magnet corresponding to a proton resonance frequency of 23.2 MHz at 32 °C. The sea cucumber was placed on NMR bed by using a 25 mm diameter radio frequency coil to collect Carr-Purcell-Meiboom-Gill (CPMG) decay signals, with 90° and 180° pulses of 13 and 26 μ s, respectively, τ -value (time between 90° and 180° pulses) of 100 μ s. Data from 8000 echoes were acquired as 8 scan repetitions. The repetition time between subsequent scans was 8 s and each measurement was performed in triplicate.

Distributed multi-exponential fitting of CPMG decay curves was performed in MultiExp Inv analysis software (Niumag Electric Corporation, Shanghai, China). The multi-exponential fitting analysis was performed on the relaxation data in the software algorithm, the SRIT analysis, to get a better fitting. From the analysis, time constants for each process were calculated from the peak position, and the area under each peak (corresponding to the proportion of water molecules exhibiting that relaxation time) was determined by cumulative integration.

2.4 MRI analysis

MRI data was also acquired on 0.5 T (permanent magnet) MiniMR-Rat (Niumag Electric Corporation, Shanghai, China) equipped with a 25 mm radio frequency coil 32 °C. T_1 and T_2 weighted images were acquired using SE imaging sequence. The following scanning protocols have been used: field of view (FOV) of 100 mm ×100 mm, slice width: 2.5 mm, slice Gap: 0.5 mm, average: 4, read size: 256, phase size: 192, T_1 weighted image echo time (TE) of 19 ms and repetition time (TR) of 300 ms, T_2 weighted image echo time (TE) of 50 ms and repetition time (TR) of 1600 ms.

2.5 Texture profile analysis (TPA)

The texture characteristics of sea cucumbers was measured using a texture analyzer (TA.XT Plus, Stable MicroSystems, London, UK) fitted with a cylindrical probe (P/50). 1.5 cm \times 1.5 cm \times 1.5 cm portions were cut from the samples with different rehydration times. The parameters were as follows: pre-speed, test speed, and post-speed were 3.0 mm/s, 1.0 mm/s and 1.0 mm/s, respectively. The time between two compressions was 5 s and the deformation ratio was 50 %. Trigger type: auto (force), trigger force: 5.0 g, data acquisition rate: 200 (pps). The tests were performed in triplicate.

2.6 Statistical analysis

Statistical analysis of PCA results was performed using Statistical Analysis System IBM SPSS statistics v19 (IBM Corporation, NY). The mean and standard deviation of the data corresponding to the elemental composition analysis, relaxation time, rehydration ratio and texture parameters were calculated. One-factor analysis of variance (ANOVA) was performed for each of the parameters. PCA was performed to show the difference in the measured variables among all the samples. Correlations between NMR parameters and rehydration ratio, texture parameters were performed. All the diagrams were plotted by Origin 8.1 software (Microcal, USA).

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3. Results and discussion

3.1 Discrimination of dried sea cucumber

Since the hydrogen protons of the lightly dried and salted dried sea cucumbers have different environments due to the different concentration of salt, the NMR could be used to discriminate the types of dried sea cucumber. Fig. 1A shows the CPMG relaxation cures for the lightly dried and salted dried sea cucumber. Generally, the trend was closely associated with the different content of water in the samples.⁸ However, it is difficult to find noticeable difference among these dried sea cucumbers from the raw data. By multiexponential fitting of the transverse relaxation data to obtain the T_2 relaxation spectra, we were able to discriminate three water populations in all sea cucumber samples (Fig. 1B). No attempt has been made to assign these peaks to the various sea cucumber components, though the dominant peak at ca. 30 ms probably arises from water protons. Relaxation time T_{21} of the lightly dried sea cucumber was about 0.32 ms, while that of the salted dried sea cucumber was about 0.21 ms (Table 1). The area of the component $T_{21}(A_{21})$ of the lightly dried sea cucumbers was about 71.57 and that of the salted dried sea cucumber was about 41.58 (Table 1). The relaxation times T_{21} and the A_{21} between the lightly dried and salted dried sea cucumbers were statistically difference (P \leq 0.05). As reported in previous study, high NaCl concentrations would result in protein denaturation, further reducing the number of macromolecules available as water-binding sites.¹⁹ In this study, the decrease in A_{21} of the salted dried sea cucumber suggests that as the proteins became denatured, less hydrophilic groups were available for water-binding. The values of T_{22} and T_{23} as well as their respective populations A_{22} and A_{23} between the lightly dried and salted dried sea cucumbers were not significantly different in this study. Therefore, statistical techniques are required to extract useful information from the T_2 decays for better discrimination of the lightly dried and salted dried sea cucumbers.



Fig. 1 The CPMG signals (A) and T_2 relaxation spectra (B) acquired from the raw data of the lightly dried and salted dried sea cucumber.

Table 1. NMR parameters obtained from the lightly dried and salted dried sea

cucumbers.						
Sea	T_{21}	T_{22}	T_{23}	4	4	4
cucumber	(ms)	(ms)	(ms)	121	A22	Z123
Lightly	0.32±0	4.51±3	31.19±	71.57±	2.81±1	2.51±1
dried	.05 ^a	.46 ^a	7.05 ^a	6.89 ^a	.57 ^a	.55 ^a
Salted	0.21±0	3.47±1	$28.03 \pm$	41.58±	1.99±1	4.15±2
dried	.01 ^b	.20ª	10.29 ^a	5.48 ^b	.08 ^a	.51ª
Different letters in a column indicate significant differences ($p < 0.05$) within						
each treatment (ANOVA)						

Principle component analysis (PCA) is a powerful statistical technique for compression of large multivariate data sets such as spectral information, which is the fundamental method used in chemometrics based on vector algebra.²² PCA was used in

this study to discriminate the lightly dried and salted dried sea cucumbers before rehydration process. The purpose of PCA method was to reduce the dimensions of NMR data set with a large number of inter-correlated variables, while retained as much of the information present in the original data as possible. The raw CPMG magnetization decays consisting of 200 spectral data points were analyzed by the PCA in order to visualize the difference between the lightly dried and salted dried sea cucumber. Fig. 2 shows the score plot of the first two principle components of PC1 versus PC2. The PCs are a set of orthogonal variables describing the variance in data. Only the first few of them can retain most of variation in describing the systematic information of all the original variables.¹³ The PC1 explained 82.3% of the total variation and discriminated clearly according to different salt content, while PC2 explained 5.1% of the total variation mainly due to the variation between replicates.(Fig. 2) Totally, the first two PCs explained 87.4% of variation in all measured variables. The results indicated that the lightly dried and salted dried sea cucumber samples were clearly identified by using NMR combined with PCA technique.



Fig. 2 Principal components PC1 and PC2 scores plot from principal component analysis.

3.2 Analysis of the rehydration process of sea cucumber

The rehydration process of the lightly dried cucumber included two steps: presoaking and repeated soaking process. First, the T_2 relaxation time spectra were measured to analyze the presoaking process. As shown in Fig. 3, the transverse relaxation curves were well described by four separate peaks, which changed along with the increase of presoaking time. Characteristic relaxation times were in good agreement with discrete results of the four components. Thus the water uptake of the lightly dried cucumber can be described by the T_2 relaxation time spectra. The population with the shortest relaxation time, T_{21} , between 1 and 2.5 ms, was considered to be the cell wall protons.²⁰ The intermediate population with a time constant of 8-15 ms (T_{22}) represented water located within highly organized protein structures and water in tertiary and quaternary protein structures.²³ The third population, T_{23} , with the relaxation time, 30-100 ms, corresponded to extramyofibrillar water.24 The component with longest relaxation time, T_{24} , with the relaxation time, 450-610 ms, was ascribed to vacuolar water.²⁰ It can be seen that the T_2 relaxation components (T_{23}) significantly increased, indicating the obvious absorption of water during the presoaking process. The T_2

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relaxation curves for 24 h and 28 h was overlapped, which demonstrated that 24 h is enough for the presoaking process.



Fig.3 T_2 relaxation spectra of the lightly dried sea cucumber for different presoaking times.

In order to further analyze the presoaking process, MRI was also used for non-invasively visualize the internal structure of the rehydrated sea cucumber, which has been recently considered as a rapid, direct, accurate and non-destructive method for food process analysis.²¹ MRI not only can determine the water distribution in food sample, but also visualize the internal structure change during the food process. Fig. 4 A shows that the T_1 and T_2 weighted images of lightly dried sea cucumber underwent presoaking process for as long as 28 h. It is of special interest to observe that the pseudo-color pictures more intuitively show the difference of each sample in layers (high proton density, red color) and core (low-high proton density, blue color). The outer layers of the sea cucumbers give a bright contrast enhancement, while the core has low signal intensity due the low water content. As we know, T_1 and T_2 weighted images can highlight the signal of different phases of water in the food samples. T_1 weighted images can visually show the bound water distribution and T_2 weighted images the free water distribution in food sample.²⁵ At preliminary stage, the maps of the lightly dried sea cucumber demonstrated that the absorbed water was mainly bound water $(T_1 \text{ weighted images})$. At about 8 h, the free water was starting to appear as shown by the T_2 weighted images. The free water continuously increased after 8 h until 24 h. After rehydration for 24 h, the water distribution became relatively uniform throughout the lightly dried sea cucumber. Quantitatively, the relative intensity of the T_1 and T_2 weighted images displayed in Fig. 4B clearly showed that the intensity of the MR images increased until 24 h and then decreased at 28 h. These results revealed that the appropriate presoaking time for the lightly dried sea cucumbers was 24 h, which was well consistent with that of the transverse relaxation curve analysis.



Fig. 4 T_1 and T_2 weighted images (A) and the corresponding histogram of relative intensity of the T_1 and T_2 weighted images (B) of the lightly dried sea cucumbers for different presoaking times.

After presoaking process and boiling for 30 minutes, the sea cucumbers underwent further rehydration process for 24, 48, 72, 96, 120 and 144 h, respectively. The water mobility and structure change during the rehydration process were analyzed by the NMR and MRI. Fig. 5 shows the representative distribution of NMR T_2 relaxation spectra containing three peaks. These peaks were identified as T_{21} , T_{22} , and T_{23} according to previous assignments. During the rehydration process, all the relaxation components $(T_{21}, T_{22} \text{ and } T_{23})$ shifted to the longer relaxation time. The T_{23} and T_{24} populations in Fig. 3 probably were merged into one peak of T_{23} in Fig. 5. Thus, we could use three components to describe the relaxation decay of water in rehydrated sea cucumbers. As shown in Table 2, T_{21} , T_{22} and T_{23} continuously increased as rehydration times' increased, in which the T_{23} differed significantly from each other (p<0.05) (Table 2). This revealed that water mobility was increasing as rehydration time increase. The T_{23} population (A_{23}) commonly described more than 90 % of the water in the rehydrated sea cucumbers (Table 2). The amplitude of water population (A_{21}) was decreasing and A_{22} and A_{23} were increasing at rehydration for 96 h, but they were decreasing after 120 h rehydration (p<0.05) (Table 2). The peak of A_{23} appeared at 96 h, indicating that 96 h was probably enough for the rehydration of lightly dried sea cucumbers.



Fig. 5 T_2 relaxation spectra of the lightly dried sea cucumber for different rehydration times. Inset shows the enlarged relaxation time T_{21} .

Table 2. NMR parameters obtained in lightly dried sea cucumber for different rehydration time.

renyuratio	on time.					
Time (h)	T_{21}	T_{22}	T_{23}	A_{21}	A_{22}	A ₂₃
24	3.97 ^a	19.00 ^a	156.71ª	74.47 ^a	518.07 ^a	8024.22 ^a
48	3.79 ^a	24.33 ^b	182.19 ^b	61.19 ^{ab}	768.70 ^b	11889.06 ^b
72	3.82 ^a	26.23°	200.73°	48.81 ^b	829.41°	12386.70 ^c
96	5.95 ^b	29.22 ^{de}	243.65 ^d	11.20 ^c	856.72 [°]	14192.92 ^d
120	8.01 ^b	28.60 ^d	259.91°	21.78 ^c	794.84 ^b	13888.12 ^e
144	7.52 ^b	29.85 ^e	286.36^{f}	10.96 ^c	777.98 ^b	13513.34 ^f
D:ff-ment	1.44.000	l	. linete sime	.: C	· · · · · · · · · · · · · · · · · · ·	0.05)

Different letters in a column indicate significant differences (p<0.05) within each treatment (ANOVA).

Fig. 6 A shows the pseudo-color pictures (low-high proton density, blue-red color) of the T_1 and T_2 weighted images of the rehydrated sea cucumber at different time points. Since the T_2 weighted images highlighted the free water distribution in the sample, it demonstrated that a great amount of water population was the free water population during rehydration process. The proportion of bound water population was much smaller by the comparison of the relative values of T_1 and T_2 in Fig. 6 B. It was likely that the presence of a larger extracellular space, facilitating the water ponetration into the enlarged space and the

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increase in the extracellular water content. After rehydration for 120 h, the water population of bound water and free water decreased (Fig. 6 B). This result was consistent with the previously mentioned A_{22} and A_{23} (Table 2). It is possible to infer that too much rehydration time will lead to formation of excessive extracellular space, thus reducing water holding capacity of the dehydrated sea cucumbers. Therefore, 96 h is enough for rehydration of the lightly dried sea cucumbers.



Fig. 6 T_1 and T_2 weighted images (A) and the corresponding histogram of relative intensity of the T_1 and T_2 weighted images (B) of lightly dried sea cucumber for different rehydration times.

3.3 Relationship between TPA and NMR

As a fast and non-destructive analytical technique, NMR can in many cases replace the more time consuming and sample destructive analysis methods if strong correlations between the NMR parameters and relative physicochemical parameter is found. Texture is another important parameter in sea cucumbers quality. In this regard, the relationship between the NMR relaxation parameters and TPA parameters including hardness, chewiness, cohesiveness, springiness and rehydration ratio of the lightly dried sea cucumbers was investigated. Table 3 shows the result of TPA parameters (hardness, chewiness, cohesiveness, springiness and rehydration ratio) of the rehydrated sea cucumber at different time points. It was noted that the TPA hardness and chewiness decreased with increasing time of rehydration (p < 0.05). The main reason for this phenomenon was that increasing time of rehydration could lead to the space larger of the fiber bundle. The rehydration increased the water-holding capacity, subsequently decreased the protein-protein interaction and increased the solubility of myofibrillar proteins, which decreased the cohesion and increased the elasticity of the muscle tissue. Moreover, the cohesiveness did not significantly decrease at the first 94 h rehydration, but displayed a significant decrease when the sea cucumber was rehydrated for 144 h. This reason probably was because at preliminary stage of rehydration, the lightly dried sea cucumber formed more compact crosslinked network structure. After rehydration for 144 h, crosslinked network structure might suffer damages by the repeated rehydration process, leading to the significant decrease of cohesiveness. However, the springiness was observed to show a significant increase after 96 h rehydration, probably due to the rehydration unfolding the protein-protein fibers, subsequently increasing the elasticity of the sea cucumber. Prolonging the rehydration time will decrease the springiness. In addition, the rehydration

ratio increased at the first 96 h of rehydration, and did not change much in the remaining rehydration time.

Table 3. Result of TPA parameters (hardness, chewiness, cohesiveness, springiness and rehydration ratio) of lightly dried sea cucumber for different rehydration times.

Time	Hardnes	Chewine	Cohesiv	Springin	Rehydrat
(h)	s (g)	SS	eness	ess	ion ratio
24	866.77 ^a	991.49 ^a	0.96 ^{ab}	1.20 ^a	8.76
48	767.62 ^b	767.13 ^b	1.00^{b}	1.00^{a}	10.32
72	450.20 ^c	449.85°	1.00 ^b	1.00 ^a	10.68
96	238.05 ^d	394.62°	0.97^{ab}	1.71 ^b	11.58
120	203.27 ^{de}	203.10 ^d	0.95^{ab}	1.00^{a}	11.35
144	165.37 ^e	156.42 ^d	0.93 ^a	0.99ª	11.80

To give a clearer understanding of the dynamics of rehydration process of dried sea cucumber, the comparison of the NMR data to TPA parameters was done with regard to water behavior during rehydration process. The correlations of NMR T_2 parameters and textural parameters were shown in Table 4. Good correlations between hardness and NMR T_2 parameters were observed (0.709 $\leq R^2 \leq 0.935$), which was significant except in the case of A22. The chewiness values showed similar correlation values (0.545 $\leq R^2 \leq 0.909$) that were always significantly. For different times of rehydration, we also observed a significant correlation between NMR T_2 parameters and rehydration ratio except T_{21} . The progressive increased in the fiber bundle space has been proposed as the main responsible for the changes in hardness, chewiness and rehydration ratio that take place in different times of rehydration.^{26, 27} Moreover, the cohesiveness were only correlated with the T_{21} (R²=0.651) and there's no correlation between the springiness and NMR parameters. The correlation values suggested that it might be possible to substitute the instrumental methods of hardness, chewiness and rehydration ratio by low filed NMR for monitoring the rehydration process of dried sea cucumber in a rapid and non-invasive way.

4. Conclusions

The observations reported here open a way to analyze the rehydration process of dried sea cucumbers by low field NMR and MRI. Measurements made through NMR with appropriate PCA statistical analysis can be extended to accumulate a set of data for identification of the different type of dried sea cucumber samples. The use of MRI enabled the visualization of the spatial distribution of water and internal structure change in the lightly dried sea cucumber during rehydration process. The NMR T_2 relaxation measurement revealed the water distribution analysis offered a supplemental view of the water uptake process in the lightly dried sea cucumbers during rehydration. During the rehydration process, the water distribution change could be measured by NMR. The results indicated that the proper presoaking and rehydration time was 24 and 96 h, respectively, for the lightly dried sea cucumber. TPA parameters of hardness, chewiness and rehydration ratio showed good correlations with NMR T_2 parameters for the rehydration process of the lightly dried sea cucumbers. All

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58 59 60 these results suggested that it could be possible to use NMR and MRI to monitor the rehydration process of dried sea cucumber and evaluate the quality of sea cucumbers.

Table 4. Linear regression analyses between NMR parameters and hardness, springiness, cohesiveness, chewiness and rehydration ratio.

<u>T</u>	'n		D			
T ₂ parameters	K	K	P			
Correlation with ha	ardness	0.700	D <0.05			
<i>I</i> ₂₁	0.842	0.709	P<0.05			
T_{22}	0.945	0.893	P<0.05			
T_{23}	0.956	0.913	P<0.05			
A_{21}	0.967	0.935	P<0.05			
A_{22}	0.715	0.511	ns			
A_{23}	0.880	0.775	P<0.05			
Correlation with spri	inginess					
T_{21}	0.006	—	ns			
T_{22}	0.113	0.013	ns			
T_{23}	0.029	0.001	ns			
A_{21}	0.296	0.088	ns			
A_{22}	0.128	0.016	ns			
A_{23}	0.142	0.020	ns			
Correlation with coh	esiveness					
T_{21}	0.807	0.651	P<0.1			
T_{22}	0.311	0.097	ns			
T_{23}	0.652	0.425	ns			
A_{21}	0.544	0.296	ns			
A_{22}	0.196	0.038	ns			
A_{23}	0.148	0.022	ns			
Correlation with che	Correlation with chewiness					
T_{21}	0.828	0.686	P<0.05			
T_{22}	0.948	0.899	P<0.05			
T_{23}	0.954	0.909	P<0.05			
A_{21}	0.913	0.834	P<0.05			
A22	0.738	0.545	P<0.1			
A23	0.887	0 787	P<0.05			
Correlation with rehydration ratio						
T_{21}	0 728	0.530	ns			
T ₂₂	0.998	0.997 P < 0.0				
T 22 T 22	0.921	0.848	P<0.05			
1 23	0.921	0.886	P < 0.05			
2121 4	0.241	0.000	r < 0.03			
A ₂₂	0.86/	0.751	P<0.05			
A_{23}	0.974	0.949	P<0.05			

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Notes and references

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- 1. S. Bordbar, F. Anwar and N. Saari, Mar Drugs, 2011, 1761-1805.
- 2. S. W. Purcell, PLoS One, 2014 9, e95075.
- Y. Bai, M. Qu, Z. Luan, X. Li and Y. Yang, LWT Food Sci Tech, 2013, 54, 570-576.
- I. S. Saguya, A. Marabia and R. Wallachb, Trends Food Sci Tech, 2005, 16, 495-506.
- 5. A. Marabi and I. S. Saguy, J Sci Food Agric 2004, 1105-1110.
- X. Duan, M. Zhang, A. S. Mujumdar and S. Wang, J Food Eng, 2010, 96, 491-497.
- M. F. Marcone, S. Wang, W. Albabish, S. Nie, D. Somnarain and A. Hill, Food Res Int, 2013, 51, 729-747.
- H. T. P. Signe M Jepsen, Søren B Engelsen, J Sci Food Agr, 1999, 79, 1793-1802.
- C. D. S. Carneiro, E. T. Mársico, R. D. O. R. Ribeiro, C. A. Conte Júnior, T. S. Álvares and E. F. O. De Jesus, J Food Process Eng, 2013, 36, 492-499.
- L. Zhang and M. J. McCarthy, Postharvest Bio Tech, 2012, 67, 96-101.
- R. d. O. R. Ribeiro, E. T. Mársico, C. d. S. Carneiro, M. L. G. Monteiro, C. A. Conte Júnior, S. Mano and E. F. O. de Jesus, LWT -Food Sci Tech, 2014, 55, 90-95.
- P. M. Santos, C. C. Corrêa, L. A. Forato, R. R. Tullio, G. M. Cruz and L. A. Colnago, Food Control, 2014, 38, 204-208.
- 13. X. Shao and Y. Li, Food Bioprocess Tech, 2010, 5, 1817-1823.
- D. W. Tang, L. K. Fellows, D. M. Small and A. Dagher, Physiology & behavior, 2012, 106, 317-324.
- S. Nakano, J. Kousaka, K. Fujii, K. Yorozuya, M. Yoshida, Y. Mouri, M. Akizuki, R. Tetsuka, T. Ando, T. Fukutomi, Y. Oshima, J. Kimura, T. Ishiguchi and O. Arai, Breast cancer research and treatment, 2012, 134, 1179-1188.
- R. A. Prestes, L. A. Colnago, L. A. Forato, L. Vizzotto, E. H. Novotny and E. Carrilho, Anal Chim Acta 2007, 596 325–329.
- 17. K. K. Patel, M. A. Khan and A. Kar, J Food Sci Tech, 2013.
- D. Fan, S. Ma, L. Wang, H. Zhao, J. Zhao, H. Zhang and W. Chen, Carbohydrate polymers, 2013, 97, 406-412.
- C. K. McDonnell, P. Allen, E. Duggan, J. M. Arimi, E. Casey, G. Duane and J. G. Lyng, Meat science, 2013, 95, 51-58.
- 20. X. Shao and Y. Li, Food Bioprocess Tech, 2013, 6, 1593-1599.
- J. Liu, K. Zhu, T. Ye, S. Wan, Y. Wang, D. Wang, B. Li and C. Wang, Food Res Int, 2013, 51, 437-443.
- L. N. L. Munck, S.B. Engelsen, R. Bro, C.A. Andersson, Chemometr Intell Lab Syst, 1998, 44, 31-60.
- I. Sanchez-Alonso, I. Martinez, J. Sanchez-Valencia and M. Careche, Food chemistry, 2012, 135, 1626-1634.
- I. G. Aursand, L. Gallart-Jornet, U. Erikson, D. E. Axelson and T. Rustad, J Agr Food Chem, 2008, 56, 6252-6260.25. Y. S. Hong, J. H. Cho, N. R. Kim, C. Lee, C. Cheong, K. S. Hong and C. H. Lee, Food chemistry, 2009, 112 267-272.
- A. Marabi, U. Thieme, M. Jacobson and I. S. Saguy, J Food Eng, 2006, 72, 211-217.
- K. A. Thorarinsdottir, S. Arason, S. Sigurgisladottir, V. N. Gunnlaugsson, J. Johannsdottir and E. Tornberg, Food chemistry, 2011, 126, 109-115.

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NMR and MRI were employed to analyze the water uptake and distribution during rehydration processing of the lightly dried sea cucumber. Good linear correlations were observed between the NMR parameters and texture profile analysis.