Ex vivo ^{1}H magnetic resonance spectroscopy of intact salmon muscle via Hadamard-encoded intermolecular multiple-quantum coherence technique

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Target audience

The target audience of present study is basic scientists and scientists who are interested in NMR study on fish quality and nutrition.

Purpose

The demand for fish product is constantly increasing and the industry of seafood is becoming one of the biggest businesses in the world. Both lipids and small metabolites are related to the nourishment and texture of fish, so it would be of great value if they can be identified and quantified. NMR spectroscopy provides a non-invasive, non-destructive, safe and pollution-free tool and has become an important analytical technique in food science. However, it is hard for traditional methods to obtain high-resolution (HR) NMR spectrum of intact fish tissues due to the intrinsic inhomogeneity of fish tissues. Nowadays, there are normally two methods for HR NMR spectrum of fish: liquid NMR of extraction and magic angle spinning NMR of *ex vivo* tissues. Recently, intermolecular multiple-quantum coherences (iMQCs) originated from dipole-dipole interactions among spins in different molecules has been utilized to obtain HR NMR spectra in the presence of magnetic field inhomogeneities. In this work, we introduce iMQC NMR technique to study intact fish muscle.

Methods

The extract of white muscle of Norwegian salmon and an intact white muscle of the same fish were studied using the Hadamard-encoded intermolecular double-quantum coherence (iDQC) method proposed by our group.[1] Some traditional methods were also employed for comparison.

All experiments were performed on a 500 MHz NMR spectrometer at 298 K using a 5 mm indirect detection probe equipped with three dimensional gradient coils. The extract was extracted from 600 mg Norwegian salmon white muscle and then dissolved in 600 μL H_2O. The intact salmon muscle was stuffed in a 5 mm NMR tube without any pretreatment. For comparison, conventional one-dimensional (1D) ^{1}H NMR and spin-echo (SE) experiment which has been used to get the spectra of intact salmon muscle were also performed.[2]

Results and discussion

The experimental results of the extract are shown in Fig. 1. The conventional 1D spectra obtained in a homogeneous magnetic field and in an inhomogeneous magnetic field with a line-width of 280 Hz are shown in Fig. 1a and 1b respectively. Clearly, the peaks are overlapped and no useful spectral information can be obtained from Fig. 1b. The Hadamard-encoded iDQC 1D spectrum was acquired in the same inhomogeneous field. The spectral line-width is reduced from 280 to 39 Hz, and the peaks become distinguishable. Most of the metabolites signal peaks appear. The experimental results of the *ex vivo* salmon muscle tissue are shown in Fig. 2. The conventional 1D spectrum in an inhomogeneous field with a line-width of 240 Hz is shown in Fig. 2b. Hardly any spectral information can be obtained from this spectrum because of the intense water signal and line broadening caused by inhomogeneous field. Even after good shimming, the resolution is still poor (Fig. 2a). The SE spectrum acquired in the well-shimmed magnetic field (a line-width of 35 Hz) is shown in Fig. 2c. Compared to Fig. 2a, more small metabolites can be observed, but only a little information of fatty acids can be obtained. In Fig. 2d, information of fatty acids is gone as well as small metabolites. The Hadamard-encoded iDQC spectrum obtained in the inhomogeneous field is shown in Fig. 2e. The water signal is effectively suppressed and most of the fatty acids signals are observable as well as the small metabolites. The relative signal intensity is slightly different from conventional SQC experiment because of the obvious different relaxation times between the metabolites and fatty acids, which also happens in SE experiment.

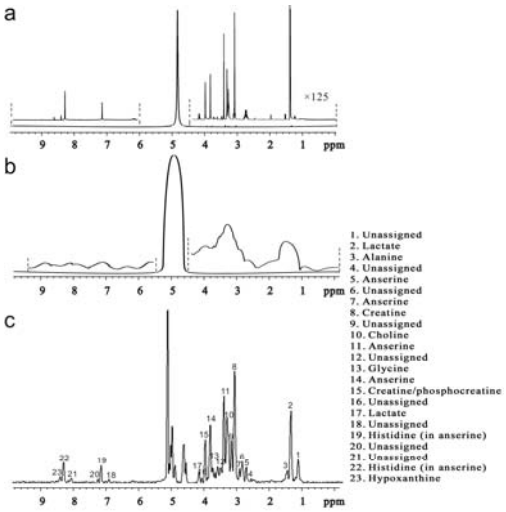

FIG. 1. ^{1}H NMR spectra of aqueous extract from white muscle of Norwegian salmon. (a) Conventional 1D spectrum in a well-shimmed magnetic field; (b) conventional 1D spectrum in a deshimmed inhomogeneous field with a line-width of 280 Hz; (c) Hadamard-encoded iDQC 1D spectrum in the same inhomogeneous field.

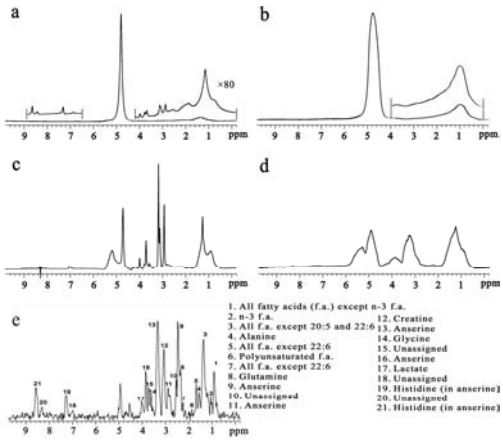

FIG. 2. *Ex vivo* ^{1}H NMR spectra of intact white muscle of Norwegian salmon. (a) Conventional 1D spectrum in a well-shimmed magnetic field; (b) conventional 1D spectrum in a deshimmed inhomogeneous field with a line-width of 240 Hz; (c) spectrum obtained using a SE sequence in the well-shimmed magnetic field; (d) spectrum obtained using a SE sequence in the same inhomogeneous field; and (e) Hadamard-encoded iDQC 1D spectrum in the same inhomogeneous field.

Conclusion

In this work, Hadamard-encoded iDQC method was employed to fast obtain high-resolution 1D NMR spectra of intact salmon muscle. This method can not only recover the spectral information concealed by inhomogeneous line broadening, but also improve the acquisition efficiency relative to conventional iMQC method.

Acknowledgement

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