

# The Application of Nuclear Magnetic Resonance Technology in Study of Concrete Materials

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## Abstract

As one of the most widely used structural materials in modern society, the optimization and improvement of concrete performance has always been a hot topic in material science research. As a non-destructive analysis method, nuclear magnetic resonance ( NMR ) technology has shown unique advantages in the study of concrete materials because it can provide material microstructure and dynamic information. This paper reviews the application of nuclear magnetic resonance technology in the hydration process, pore structure analysis and durability evaluation of concrete materials, and discusses the application progress of nuclear magnetic resonance technology in the quantitative analysis of cement-based materials. By analyzing the advantages and limitations of different nuclear magnetic resonance techniques, it provides theoretical basis and technical support for further research and engineering application of concrete materials.

## Keywords

Concrete, nuclear magnetic resonance, solid-state nuclear magnetic resonance, pore structure.

## 1. Introduction

As a heterogeneous, multi-phase and multi-level composite system, concrete has extremely complex microscopic pore structure [1]. Its quality has a crucial impact on the strength and durability of buildings. Therefore, the detection and evaluation of concrete quality is essential. Various techniques have been used to study the pore characteristics of concrete materials, such as mercury intrusion method [2], scanning electron microscope (SEM) [3], gas adsorption method [4], NMR and small angle X-ray scattering method [5]. Different test methods have different pore size test ranges and accuracy.

In recent years, NMR technology has been introduced into the field of concrete quality testing. By detecting the parameters such as moisture and pore structure in concrete, the quality of concrete can be comprehensively and accurately evaluated. NMR technology can accurately obtain pore characteristics by measuring the transverse relaxation time of hydrogen atoms in porous materials, so as to intuitively quantify and qualitatively analyze the microstructure inside porous materials [6], and has the advantages of non-destructive testing and continuous monitoring. Compared with NMR technology, other test methods have limitations such as only qualitative analysis of the geometry and distribution of pores [7], limited pore size test range and destruction of the original pore structure [8], which affect the characterization of pore structure characteristics of porous materials. As an effective method for non-destructive quantitative analysis of pore structure, NMR technology has been widely used in the characterization of microscopic pore structure of rock and concrete [9, 10, 11]. In this paper, the main application of NMR technology in the study of concrete materials and its future development prospects are reviewed.

## 2. Rationale

NMR is a physical phenomenon, in which some nuclei have non-zero magnetic moments. Under the action of an external magnetic field, the magnetic moments of these nuclei can absorb electromagnetic radiation at a specific frequency, thereby achieving energy level transitions. This phenomenon can be used to study the structure and dynamic characteristics of molecules. The basic principle of NMR involves the spin of the nucleus, which produces a magnetic moment. When placed in an external magnetic field, the magnetic moment aligns with the magnetic field and produces different energy levels. The transitions between these energy levels can be achieved by absorbing electromagnetic waves at a specific frequency, which is called the Larmor frequency. In the NMR experiment, the nucleus is excited by applying a radio frequency pulse, and then the signal emitted during the relaxation process is measured to obtain information about the structure and dynamics of the sample.

## 3. Application Technology of NMR in Concrete

### 3.1. NMR Hydrogen Spectrum Test Technology

NMR spectroscopy ( $^1\text{H-NMR}$ ) is an application of the NMR effect of hydrogen-1 in the molecule in nuclear magnetic resonance spectroscopy. It can be used to determine the molecular structure. When the sample contains hydrogen, especially the isotope hydrogen-1, nuclear magnetic resonance spectroscopy can be used to determine the structure of the molecule. The hydrogen-1 atom is also called protium.

$^1\text{H-NMR}$  technology is widely used in concrete testing. It provides an in-depth insight into the microstructure and hydration behavior of concrete in a non-destructive manner. Kurihara et al. [12] used  $^1\text{H-NMR}$  relaxation technique to evaluate the surface area of cement and compared it with the BET surface area measured by water vapor. It was found that the total surface area evaluated by  $^1\text{H-NMR}$  was always 2-2.5 times the BET surface area of water vapor. Glawe et al. [13] used non-destructive unilateral hydrogen nuclear magnetic resonance to determine the pore structure change of cement-based materials and the carbonization degree of mortar. In terms of quality control and defect detection,  $^1\text{H-NMR}$  technology can reveal problems such as holes, cracks and uneven hydration. It can also monitor the aging and degradation of concrete under the action of the environment and provide data support for the long-term performance evaluation of materials. [14] With the development of new concrete materials, the importance of  $^1\text{H-NMR}$  technology in materials science research is increasing, and it also plays an important role in the field of education and training to help professionals better understand the complex properties of concrete.

### 3.2. Solid State Nuclear Magnetic Resonance Testing Technology

Solid State Nuclear Magnetic Resonance (SSNMR) is an analytical technique based on solid samples. It has a strong adaptability to the state of the sample, which can be used for the structural analysis of solid materials with high crystallinity, as well as the structural analysis of solid materials and amorphous materials with low crystallinity. It studies the different local environments around various nuclei, that is, the short- and medium-range interactions, and complements the methods of X-ray diffraction, neutron diffraction, and electron diffraction to study the long-range overall structure of solids. Especially in the study of amorphous, because there is no long-range order, NMR is more important, so it is gradually becoming a conventional means of structural analysis of amorphous materials [15].

At present, solid-state NMR mainly uses magic angle rotation technology, cross-polarization technology and rotating sideband suppression technology to detect specific nuclei such as  $^{13}\text{C}$ ,  $^{29}\text{Si}$ ,  $^{27}\text{Al}$  and  $^{31}\text{P}$ . Among them, magic angle rotation technology can eliminate the spectral line

broadening effect caused by dipole coupling interaction and chemical shift anisotropy. Cross-polarization technology can improve the detection sensitivity of dilute nuclei, and rotating sideband suppression technology eliminates the rotating sideband generated by rapid rotation of samples [16]. Therefore, in order to narrow the spectral line, increase the sensitivity, and obtain high-resolution solid-state NMR spectra, several techniques such as CP, MAS and TOSS are usually used in combination for detection [17] to obtain richer information about the different chemical environments of the nucleus, the relative content of the corresponding groups, and the molecular motion.

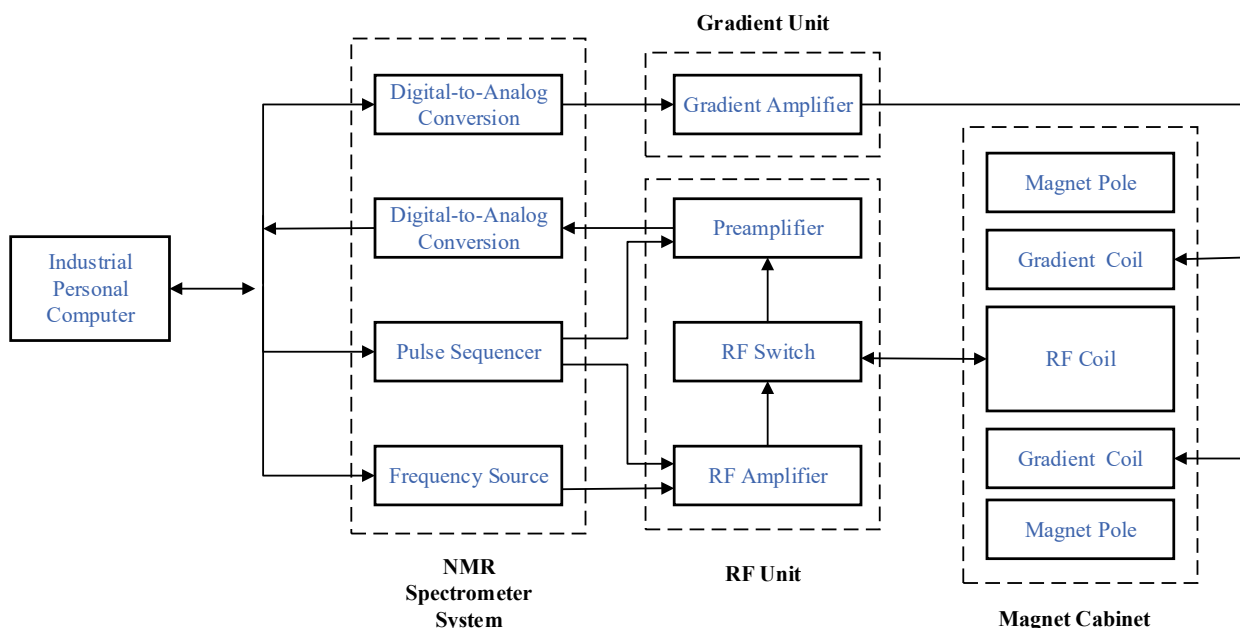
SSNMR technology has important applications in the study of cement concrete materials. Wang et al. [18] studied the effect of silicon powder on the C-S-H structure by using solid high-resolution  $^{29}\text{Si}$  NMR technology and Fourier infrared spectroscopy. From the micro level, it was found that the anti-abrasion performance of concrete can be effectively improved after adding silicon powder. Wang [19]. studied the fraction of  $^{29}\text{Si}$  in silicates in different chemical environments using solid-state NMR techniques. Méducin et al. [20] studied the microstructure of cement hydration process under high temperature and high pressure. High-resolution solid-state  $^1\text{H}$  and  $^{29}\text{Si}$  NMR was used to observe the characterization of magnesium hydroxide and carbomagnesite.

Rottstegge et al. [21] used SSNMR to study the effects of different organic and inorganic additives on the hydration and hardening of cement at the nanoscale. It was found that a small amount of additives had a strong effect on the hydration and hardening process and crystallization process of cement. The results at the molecular level provided by NMR are related to the changes in the mechanical properties of cement pastes. Huang et al. [22] used NMR technology to analyze the pore characteristics of basalt-polypropylene hybrid fiber reinforced concrete. The results show that the bonding state and distribution of fibers in the concrete matrix are the main reasons affecting the fractal characteristics of HBPRC pores. The distribution and connectivity of concrete pores can be directly obtained by nuclear magnetic resonance technology, which provides a reference for the design and reinforcement of concrete. SSNMR is used to study the hydration process of cement, analyze hydration products, pore structure and water distribution, analyze the chemical composition of hydration [23], detect the chemical shift of materials [24], and develop and optimize admixtures and functional additives. These studies are helpful to understand and optimize the performance of concrete and promote the development and engineering application of new materials.

### 3.3. Low-field Nuclear Magnetic Resonance Testing Technology

Low-Field Nuclear Magnetic Resonance ( LF-NMR ) is a nuclear magnetic resonance ( NMR ) technique that is performed at low magnetic field strength, usually in the range of a few millitesla to a few hundred milli-tesla. Low field nuclear magnetic resonance ( NMR ) uses nuclear magnetic resonance ( NMR ) phenomena, that is, energy level splitting and energy absorption and release of nuclei under the action of an external magnetic field. By measuring the longitudinal relaxation time (  $T_1$  ) and transverse relaxation time (  $T_2$  ) of the hydrogen nucleus in the sample, the pore structure, water state and molecular motion of the sample can be analyzed.

The function of low field nuclear magnetic resonance instrument is divided into five parts : industrial personal computer, spectrometer system, gradient unit, radiofrequency ( RF ) unit, magnet cabinet. The architecture diagram is shown in Fig 1.



**Figure 1.** Nuclear magnetic resonance instrument architecture diagram

### 3.3.1. Nuclear magnetic resonance $T_1$ spectrum test technology

Nuclear magnetic resonance  $T_1$  spectroscopy is a method for measuring the longitudinal relaxation time ( $T_1$ ) of hydrogen nuclei in samples. The  $T_1$  relaxation time reflects the spin-lattice relaxation process, that is, the time constant of the sample from the excitation state to the equilibrium state. This technique can provide the physical properties of the sample, such as pore structure, fluid properties, etc. Through the  $T_1$  spectrum, the pores of different sizes can be more accurately distinguished, the development of microcracks can be evaluated, and the porosity and pore size distribution can be accurately quantified, providing accurate data support for the durability evaluation of concrete (such as impermeability and freeze-thaw resistance).

Faedis et al. [25] studied the effect of silicate clay on cement hydration through the  $T_1$  relaxation time of water protons during hydration, and found that clay had an effect on the compressive strength of the material. She et al. [26] used low-field nuclear magnetic resonance technology to observe the  $T_1$  relaxation time and studied the hydration process of cement pastes with different water-cement ratios and superplasticizers. Faure et al. [27] used the time evolution of NMR  $T_1$  relaxation time distribution to determine the water transfer in cannabis lime concrete. Pang et al. [28] used the  $^1\text{H}$  spin-lattice relaxation method ( $T_1$ , longitudinal) to study the cement paste with superplasticizer. The results showed that polycarboxylate superplasticizer could prolong and reduce the main peak of  $T_1$  relaxation, which revealed the interference of dispersion mechanism on the evaporated water during cement hydration.

With the development of technology,  $T_1$  spectrum test technology can describe the pore structure of concrete in detail and accurately at the micro scale [29], so as to provide more accurate data support for the durability evaluation of concrete.

### 3.3.2. Nuclear magnetic resonance $T_2$ spectrum test technology

Nuclear magnetic resonance  $T_2$  spectrum test technology is a technology based on the principle of nuclear magnetic resonance, which is specially used to analyze the distribution state and pore structure characteristics of fluids (such as water and oil) in porous media.  $T_2$  spectrum, namely transverse relaxation time spectrum, in nuclear magnetic resonance technology,  $T_2$  describes the decay rate of transverse magnetization vector  $M_{xy}$ , and the time required for

transverse magnetization vector to decrease from the maximum value to 37% of the maximum value is defined as  $T_2$  [30].

He et al. [31] proposed a new method to construct capillary pressure curve by using nuclear magnetic resonance  $T_2$  distribution. Through comparative analysis, it is proved that this method can effectively improve the structural accuracy of capillary pressure curve, especially for the formation with complex pore structure. It has important application value. Wang et al. [32] proposed a nonlinear transformation method to convert the NMR  $T_2$  spectrum into a more accurate pore distribution by performing NMR and mercury injection experiments on 10 low-permeability cores. This method considers the problem of insufficient mercury intrusion saturation, and establishes the empirical formula of conversion coefficient and power law through experimental data, which effectively reflects the microscopic pore structure of the core. Bai et al. [33] quantitatively characterized the NMR  $T_2$  spectrum of sandstone rock, extracted the characteristic variables reflecting the pore structure and fluid occurrence state, and verified the reliability of these parameters in evaluating the microscopic pore structure of reservoir combined with mercury injection experimental data, which effectively improved the accuracy and operability of reservoir quality evaluation. Wang et al. [34] tested the pore structure of natural pumice lightweight aggregate concrete under freeze-thaw cycles by nuclear magnetic resonance (NMR), and obtained the porosity, the distribution of transverse relaxation time  $T_2$ , and the results of magnetic resonance imaging (MRI). It was found that with the increase of freeze-thaw cycles, the total area and porosity of  $T_2$  spectrum increased significantly. At the same time, the results of nuclear magnetic resonance detection were analyzed by ultrasonic detection technology, which verified that the new nuclear magnetic resonance technology had high accuracy and practicability in the study of freeze-thaw concrete damage. Deng et al. [35] used nuclear magnetic resonance to study the microstructure damage process of air-entrained recycled coarse aggregate concrete (ARAC) and non-ARAC concrete under freeze-thaw environment. By comparing the changes of nuclear magnetic resonance  $T_2$  spectrum with the changes of macroscopic structure, it was found that the flexural strength of recycled concrete was significantly affected by the total proportion of mesopores and macropores. Zhang et al. [36] obtained the  $T_2$  spectrum of four kinds of self-compacting concrete with different aggregates and different ages by nuclear magnetic resonance technology, obtained the pore size distribution curve, and analyzed the influence of pore structure on the mechanical properties of self-compacting concrete. The research shows that the pores with larger spectral area have a greater influence on the mechanical properties of self-compacting concrete. Wu et al. [37] tested the microstructure of polyvinyl alcohol fiber concrete under salt intrusion freeze-thaw cycle by nuclear magnetic resonance  $T_2$  spectrum, and concluded that PVA fiber concrete showed good frost resistance in the early stage of freeze-thaw cycle. Wang et al. [38] established the relationship between the strength and pore characteristics of basalt fiber concrete with different fiber contents by nuclear magnetic resonance  $T_2$  spectroscopy. The pore distribution curves of BFRC with different fiber contents were obtained by nuclear magnetic resonance  $T_2$  spectroscopy. The study found that basalt fiber can effectively improve the strength of concrete and there is a significant correlation between concrete strength and pore changes. Wang et al. [39] analyzed the  $T_2$  spectrum of alkali-activated slag foamed concrete (A-FC), and found that the compressive strength of A-FC decreased with the increase of pore size, porosity and porosity. The main reasons for the increase of strain in low-strength A-FC include bottom compression, internal stress dissipation through pore structure and change of failure mode.

Nuclear magnetic resonance  $T_2$  spectrum test technology provides a non-destructive and in-depth analysis method for evaluating and optimizing concrete performance and ensuring structural safety and durability by analyzing pore structure, water state and damage in the field of concrete.

#### 4. Deficiencies of NMR Testing Technology

NMR technology has a certain application in the field of concrete testing, but there are also some shortcomings.

(1) Test sample size limit. The test diameter of NMR equipment is usually small, which limits the test ability of large concrete samples. For large concrete structures that need to be tested, it may be necessary to cut the sample or prepare a smaller sample, which may have a certain impact on the results.

(2) Cost and operational complexity. The cost of high-performance NMR equipment is relatively high, and the purchase and maintenance require more funds. On the other hand, the operation method of NMR equipment is complex, and it is necessary to have certain professional technicians to operate and analyze data, which limits the popularization and application of NMR equipment in ordinary laboratories to a certain extent.

(3) Environmental impact. Magnetic materials such as steel bars in concrete can significantly interfere with NMR signals, resulting in inaccurate test results [40]. In the field detection, the existence of these magnetic substances is inevitable, so special designed equipment or test methods are needed to reduce the interference. In addition, the external magnetic field interference will also affect the NMR test, which requires a good shielding environment.

(4) Limitations of humidity and moisture measurement. Nuclear magnetic resonance ( NMR ) technology is a non-destructive detection method, which can measure the moisture distribution in concrete. However, NMR technology has some limitations in measuring highly wet or completely dry samples. During the curing process of concrete, the moisture content will undergo dynamic changes, which may require long-term continuous testing. In this case, NMR technology may not be able to provide stable and accurate measurement results [41]. In order to improve the accuracy and stability of the measurement, it may be necessary to combine other techniques or methods, such as using low field nuclear magnetic resonance ( LF-NMR ) to study the water evolution of concrete during standard curing, or using X-ray transmission imaging technology to track the water absorption process of concrete [42].

#### 5. Development Prospects

NMR technology has shown significant foresight and innovation in the development trend of concrete structure detection. The accuracy and versatility of nuclear magnetic resonance instruments indicate that nuclear magnetic resonance will continue to progress and expand in key dimensions in the future development.

(1) High-precision pore structure characterization. The continuous advancement of technology will promote NMR to achieve a more detailed and accurate description of the internal pore structure of concrete at the microscopic scale. Through enhanced resolution and signal processing algorithms, pores of different sizes can be more accurately distinguished, micro-crack development can be evaluated, and porosity and pore size distribution can be accurately quantified, providing more accurate data support for concrete durability assessment.

(2) Intelligent real-time monitoring system. Future NMR technology is expected to be integrated into the concrete structure monitoring system to realize real-time monitoring of the whole process from raw material ratio, pouring, curing to service period. This includes the development of embedded NMR sensors that can instantly feedback key parameters such as concrete hydration process, early strength development, and moisture state changes, providing instant information for structural health management and preventive maintenance.

(3) Innovation of portable and automated equipment. The development of portable and efficient NMR detection equipment, combined with automatic data acquisition and processing system, will greatly improve the flexibility and efficiency of on-site detection, reduce the

difficulty and cost of operation, make NMR technology closer to the actual engineering needs, and facilitate large-scale application.

(4) Advanced data analysis and artificial intelligence (AI) application. Using artificial intelligence technologies such as machine learning and deep learning to conduct in-depth analysis and pattern recognition of massive NMR data and establish prediction models can not only predict the deterioration of concrete performance in advance, but also optimize material design and construction schemes, providing scientific decision-making basis for intelligent construction.

## 6. Conclusion

As a new type of concrete quality detection method, NMR technology has broad application prospects in the field of concrete quality detection. By detecting the moisture content, pore structure and other parameters in concrete, the quality of concrete can be evaluated comprehensively and accurately. In the future, with the continuous development and improvement of NMR technology, it is believed that its application in the field of concrete quality inspection will be more widely promoted and applied.

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