

UC Irvine

UC Irvine Previously Published Works

Title

3D-printed dissolvable inserts for efficient and customizable fabrication of NMR transceiver coils

Permalink

<https://escholarship.org/uc/item/3hh829gg>

Authors

Kelz, Jessica I
Kelly, John E
Martin, Rachel W

Publication Date

2019-08-01

DOI

10.1016/j.jmr.2019.06.008

Peer reviewed



Published in final edited form as:

J Magn Reson. 2019 August ; 305: 89–92. doi:10.1016/j.jmr.2019.06.008.

3D-printed dissolvable inserts for efficient and customizable fabrication of NMR transceiver coils

Jessica I. Kelz^a, John E. Kelly^a, Rachel W. Martin^{a,b}

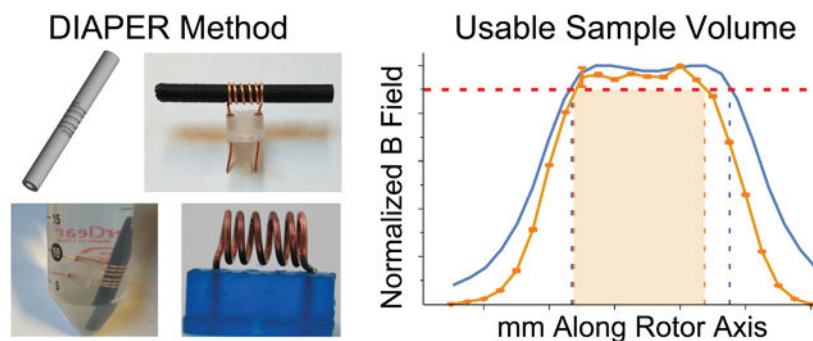
^aDepartment of Chemistry, University of California, Irvine 92697-2025

^bDepartment of Molecular Biology and Biochemistry, University of California, Irvine, 92697-3900

Abstract

We describe a simplified method for improving the reproducibility of transceiver coil fabrication for nuclear magnetic resonance (NMR) through single-use templates made from 3D-printed polymer forms. The utility of dissolvable inserts for achieving performance enhanced resonators (DIAPERs) is tested herein by a comparison of RF homogeneity along the rotor axis for variable-pitch solenoids with different inter-turn spacing. Simulated B_1 field profiles are compared to experimental homogeneity measurements, demonstrating the potential of this approach for making NMR coils quickly and reproducibly.

Graphical Abstract



Keywords

transceiver coil; NMR instrumentation; 3D printing; RF homogeneity; MAS probe; variable-pitch solenoid; magnetic field simulation

1. Introduction

Additive fabrication, or 3D printing, promises to minimize material waste and reduce the manufacturing cost of constructing intricate pieces in a variety of settings [1]. This

Publisher's Disclaimer: This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final citable form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

technology has been used to produce key parts of magic angle spinning (MAS) NMR probes, including sample exchangers [2] and stators for a spherical rotor system [3]. So far, the use of 3D printing technology to produce NMR coils and coil forms has been limited by the available materials. Available insulators are typically organic polymers or resins that produce significant proton background signals and behave as lossy dielectric materials, making them poor choices for pieces that will be in direct contact with the transceiver coil. Here we present a method for reproducibly fabricating NMR coils with particular dimensions using 3D-printed dissolvable inserts, using solenoids with different dimensions as a proof of concept.

Solenoids are arguably the best-characterized axial resonators and have been used extensively in MAS probes. They are frequently used in solid-state NMR due to their large filling factor, scalability, ease of fabrication, ability to be multiply tuned [4] and reasonable SNR [5]. Another advantage is that solenoids generate strong B_1 fields even when oriented at the magic angle. In theory, an infinitely long solenoid has perfect RF homogeneity. However, in practice solenoids with axial lengths, diameters and number of turns appropriate for use in MAS probes fall short of the optimal geometry. The generated B field along the cylinder axis drops off significantly toward the coil ends, limiting the homogeneous region to 50% or less of the available volume in many probes [6]. Several variable-pitch solenoids with decreased spacing between turns toward the ends, expanding to a maximum at the center, have been proposed to improve homogeneity and thereby increase the readily usable sample volume.

One of the first to be introduced used a modified Biot-Savart formula to vary the pitch within experimentally justifiable limits for an eight-turn coil, followed by integration to determine the magnetic field profile [7]. Another group determined the windings required to achieve a square field profile represented by an equiripple function at high frequencies [8]. Although the dimensions and frequencies differ significantly from the coils designed for this study, the authors suggest that the equiripple function has the advantage of flexibility in design. A ribbon coil of varying width has also shown improvement to radial homogeneity for higher frequencies by minimizing gaps [9]. Improvements in solenoid RF homogeneity have been augmented by computational [10] and theory-driven simulation approaches [4, 11, 12]. Here we demonstrate the construction of solenoid coils with particular dimensions, using a CAD program to design a precisely specified coil and its corresponding template. The template is then 3D printed using a dissolvable material and dissolved away in appropriate solvent after the coil is wound. This procedure was validated by comparing experimental (bench-top) and simulated B_1 field homogeneity for a constant pitch and three different variable-pitch solenoids. The primary objective of this communication is to introduce a coil fabrication method that is fast, reproducible, and allows NMR coils to be easily fabricated once an acceptable design is established.

2. Methods

2.1. Coil Designs

Four coils were designed with dimensions suitable for use in a standard solid-state NMR probe with a 3.2 mm rotor. Coils were drawn in Inventor Professional 2017 using 0.6 mm diameter (22 gauge) copper wire, each having six turns and an inner diameter of 3.8 mm.

2.2. Coil Fabrication Technique

The coils described above were built using 3D-printed polymer forms referred to herein as dissolvable inserts for achieving performance enhanced resonators (DIAPERs). The coil forms were designed in Inventor with the radius of the 22 gauge wire cut from a cylinder of diameter 4.1 mm using pitch and turn parameters listed in Table 1. In preparation for 3D printing, the cylinder was made hollow to reduce waste and expedite the removal process by increasing surface area accessible to solvent. Files were exported in STL format, compatible with Cura 3D printing software which was required for printing on the LulzBot TAZ 6 and FlashForge Creator Pro. CAD drawings and STL files are available upon request. All inserts were printed within one hour at 0.2 mm resolution using IC3D 3 mm acrylonitrile butadiene styrene (ABS) filament. Each of the four coils was hand wrapped on the template and leads positioned inside a spinning assembly coil support. ABS dissolves in acetone at room temperature, making extraction of the coils possible with minimal risk of deformation. DIAPERs were removed by soaking in approximately 20 mL of acetone in a 50 mL conical centrifuge tube stored upright overnight. Residual ABS was removed by direct application of acetone using a paintbrush. The inductance of each coil was determined by finding the slope of a curve defined by capacitance vs. frequency and is provided in Table 1. Six standard American Technical Ceramics Corp. chip capacitors were used ranging from 3.3 pF to 12 pF.

2.3. Experimental Homogeneity Measurements

To evaluate spatial homogeneity of the B_1 field along the rotor axis ball-shift assays [13] were performed in triplicate for each coil. Ball-shift assays were executed by moving a conductive ring along the rotor axis in discrete steps defined by the thread pitch, and measuring the resonance frequency shift [14]. The experimental apparatus used a 1.9 mm outer diameter, 0.3 mm thick, and 0.8 mm long copper ring on the end of a 4-40 threaded rod with an observable linear increment of 0.6 mm. A modified Varian HXY probe was tuned to 200 MHz with a minimum S_{11} of 40 dB. Shifts in the resonance frequency were recorded by observing the response on an Agilent Technologies ENA Series Network Analyzer after moving the conductor one increment at a time until a distance comparable to the rotor length had been achieved, roughly 24 turns. This process was repeated three times to ensure reproducibility with the resulting field profile generated from the average of each point normalized to the maximum frequency shift of each trial. Error was calculated by determining the standard deviation and dividing it by the square root of the number of repetitions for each axial position. Results were plotted in Wolfram Mathematica version 11.

2.4. B Field Modeling

High-frequency electromagnetic simulations were performed using Computer Simulation Technology Microwave Studio (CST MWS). Coils were imported using the IPT file format generated by Inventor. The coil material was set to the CST library value for pure copper. A discrete port was connected to one lead and defined to have 50 Ω resistance, consistent with the desired impedance of our NMR probe. A tuning capacitor was defined as a lumped element attached in parallel to the port and alternate lead. The capacitance value was determined using RLC circuit theory, $f = \frac{1}{\sqrt{LC}}$, in order to tune to 200 MHz. The excitation duration was approximately 150 ns based on a frequency range defined as ± 10 MHz around 200 MHz. Boundary conditions were defined as open add space, and background was defined as normal. Based on an estimated maximum fillable length of 15.2 mm, a line was defined along the rotor axis and centered with respect to the coil length and diameter. After running each time-domain simulation, the 1D magnetic field profile at 200 MHz was evaluated along the axial curve. Unless otherwise specified, the default mesh is used to determine the step size, however this was adjusted to match the experimental increment of 0.6 mm by using Template Based Post Processing. The results were exported as a .txt file, normalized, and plotted in Wolfram Mathematica version 11.

3. Results and Discussion

Traditionally, constant pitch solenoids are made either freehand or by wrapping two wires around a cylinder, where the wire to be removed uniformly establishes the distance between turns on the remaining coil. For this study the uniform pitch was chosen to be slightly larger than double the wire diameter so that it would produce a coil of comparable axial length to the model variable-pitch coil [15]. These coils were designed to have six turns, as this typically yields an inductance suitable for our desired frequency range [16]. Previous experimental observations indicated that the smallest allowable distance between turns without being significantly prone to arcing is roughly 0.8 mm especially at high power. Therefore, this was established as the tightest pitch in the adjusted designs for this study. The stretched variable-pitch coil was developed as a modification of the work of Idziak et al. [7], adjusted to the conserved coil parameters stated in the methods section. We chose it to test changes in number of turns between otherwise similarly dimensioned coils and the limitations of gap size between windings, whereas the differences between the other variable-pitch coils are quite subtle. The model variable-pitch coil changes the revolutions per pitch in addition to the pitch itself [15]. To control for changing the revolutions, a ratio-pitch coil was designed with comparable inter-turn spacing by increasing the pitch per one revolution based on ratios defined by the smallest pitch. In this study, the designs include one constant pitch and three different variable-pitch coils, shown in Figure 1. Detailed information for each design is provided in Table 1.

Among other factors, RF homogeneity in NMR resonators sensitively depends on symmetry, which is often difficult to achieve in hand-made fabrication. Relatively small deviations can negatively impact RF homogeneity. Therefore, realizing the performance enhancements afforded by variable-pitch coils requires optimization of spacing between turns, typically achieved by careful construction by an experienced researcher. DIAPERs provide a more

readily implementable approach that does not require extensive expertise in coil fabrication. The complete process is shown in Figure 2. Using this procedure, four verifiably different coils were created within two days, with cost of manufacture totaling under 10 dollars.

Development of this method was primarily motivated by interest in extending the utility of 3D printing for improvements in NMR instrumentation. The problem chosen for proof of concept is the comparison of variable-pitch solenoids with different dimensions with respect to RF homogeneity. Theory-driven modeling software, CST MWS, was utilized to rationalize the experimental approach through comparison to simulated results for the same coils. Figure 3 shows experimental and modeled data for each of the four coil designs. The homogeneous region in this study is defined at 90% of the normalized field magnitude.

Two of the three variable-pitch designs show improvements over constant pitch, however the stretched variable-pitch design yielded a negligible homogeneous range. It was expected that lessening the number of turns, directly increasing gap size, would yield significant field fluctuations [9], providing a dramatically different profile to test the methods of analysis. The findings demonstrate that dimensions must be considered comprehensively and that variable pitch alone does not inevitably extend the homogeneous region. Based on the measurements performed here, the model variable pitch is effectively indistinguishable from the ratio variable pitch. It would be possible to improve the resolution of this measurement by using a finer thread pitch on the ball shift apparatus, or by using the NMR signal to map the homogeneity [4, 14, 17, 18]. However, based on the CST results, the improvement to the length of the axial homogeneous region of the model variable pitch over the ratio pitch is only about 4%, which is likely to be negligible for most practical NMR applications. Use of the model variable-pitch design shows an improvement of 4.0 μL or 40% based on experimental measurements over constant pitch. The coils were designed to have nearly identical axial length; therefore improvements can be directly associated with changes in pitch.

Our model variable-pitch solenoid was originally designed to fit within a Modified Alderman-Grant Coil (MAGC) [19] in a crossed-coil probe, which limited the available axial length to 8.9 mm. If the solenoid were to be used in a single-coil design, this length could be extended to increase the viable sample volume further. Analyzing the percentage of homogeneous region to the total axial length of the coil along the rotor axis showed a 15% improvement with the model variable pitch over constant pitch. A common commercially available Revolution NMR 3.2 mm rotor that accommodates both desirable sample volume and spinning speed can hold 22.0 μL . Based on the designs in this study, constant pitch could utilize 45% of this volume, while the model variable pitch extends this to 63%. At a minimum, this offers additional flexibility in optimizing experiments. This represents a significant performance enhancement, especially for NMR experiments where sensitivity is generally a concern that can be partially mitigated by probing larger sample volumes.

4. Conclusions

Here we present a method for reproducibly making variable-pitch solenoids of particular dimensions using 3D-printed dissolvable inserts. This method enables accurate control of

pitch, verified through observable differences in magnetic field profiles. This is advantageous because accurately constructed variable-pitch solenoids can demonstrate significant improvements to axial RF homogeneity as compared to constant-pitch designs. More broadly, 3D-printed coil templates may prove to be a viable alternative to traditional fabrication methods, offering a fast, inexpensive, reproducible, easily modifiable and scalable approach for design and construction of multiple turn coils. Future work will include testing single-use DIAPERS on the production of more complex resonators such as saddle coils or other transverse resonator designs, as well as detailed investigation of the performance of these coils in NMR experiments.

Acknowledgements

This work was supported by NIH grant 2R01EY021514-06 and NSF grant CHE-1308231 to R.W.M. J.I.K. acknowledges support from NIH grant T32 GM108561 and NSF GRFP DGE-1321846. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation. The authors would like to thank the Computer Simulation Technology, GmbH for providing CST Microwave Studio that has been instrumental in this work. The authors gratefully acknowledge Filippo Capolino and Hamidreza Kazemi for insightful discussions, Ed Twehous of Revolution NMR for helpful correspondence, the Nowick lab for use of the FlashForge Creator Pro, and UCI Fabworks facility for training and access to a variety of 3D printers.

References

- [1]. Guo N, Leu MC, Additive manufacturing: technology, applications and research needs, *Frontiers in Mechanical Engineering* 8 (3) (2013) 215–243.
- [2]. Barnes AB, Mak-Jurkauskas ML, Matsuki Y, Bajaj VS, van der Wel PC, DeRocher R, Bryant J, Sirigiri JR, Temkin RJ, Lugtenburg J, Herzfeld J, Griffin RG, Cryogenic sample exchange NMR probe for magic angle spinning dynamic nuclear polarization, *Journal of Magnetic Resonance* 198 (2) (2009) 261–270. [PubMed: 19356957]
- [3]. Chen P, Albert BJ, Gao C, Alaniva N, Price LE, Scott FJ, Saliba EP, Sesti EL, Judge PT, Fisher EW, Barnes AB, Magic angle spinning spheres, *Science Advances* 4 (9) (2018) eaau1540. [PubMed: 30255153]
- [4]. Tošner Z, Pura A, Struppe JO, Wegner S, Engelke F, Glaser SJ, Reif B, Radiofrequency fields in MAS solid state NMR probes, *Journal of Magnetic Resonance* 284 (2017) 20–32. [PubMed: 28946058]
- [5]. Hoult DI, Richards RE, The signal-to-noise ratio of the nuclear magnetic resonance experiment, *Journal of Magnetic Resonance* (1969) 24 (1) (1976) 71–85.
- [6]. Gupta R, Hou G, Polenova T, Vega AJ, RF inhomogeneity and how it controls CPMAS, *Solid State Nuclear Magnetic Resonance* 72 (2015) 17–26. [PubMed: 26422256]
- [7]. Idziak S, Haerberlen U, Design and construction of a high homogeneity RF coil for solid-state multiple-pulse NMR, *Journal of Magnetic Resonance* (1969) 50 (2) (1982) 281–288.
- [8]. Leifer MC, RF solenoid with extended equiripple field profile, *Journal of Magnetic Resonance* 105 (1993) 1–6.
- [9]. Privalov AF, Dvinskikh SV, Vieth H-M, Coil design for large-volume high- B_1 homogeneity for solid-state NMR applications, *Journal of Magnetic Resonance, Series A* 123 (2) (1996) 157–160.
- [10]. Engelke F, Electromagnetic wave compression and radio frequency homogeneity in NMR solenoidal coils: computational approach, *Concepts in Magnetic Resonance* 15 (2) (2002) 129–155.
- [11]. Doty FD, Kulkarni J, Turner C, Entzminger G, Bielecki A, Using a cross-coil to reduce RF heating by an order of magnitude in triple-resonance multinuclear MAS at high fields, *Journal of Magnetic Resonance* 182 (2) (2006) 239–253. [PubMed: 16860580]

- [12]. Krahn A, Priller U, Emsley L, Engelke F, Resonator with reduced sample heating and increased homogeneity for solid-state NMR, *Journal of Magnetic Resonance* 191 (1) (2008) 78–92. [PubMed: 18187352]
- [13]. Maier LC Jr, Slater JC, Field strength measurements in resonant cavities, *Journal of applied physics* 23 (1) (1952) 68–77.
- [14]. Paulson EK, Martin RW, Zilm KW, Cross polarization, radio frequency field homogeneity, and circuit balancing in high field solid state NMR probes, *Journal of Magnetic Resonance* 171 (2) (2004) 314–323. [PubMed: 15546758]
- [15]. Collier KA, Sengupta S, Espinosa CA, Kelly JE, Kelz JI, Martin RW, Design and construction of a quadruple-resonance MAS NMR probe for investigation of extensively deuterated biomolecules, *Journal of Magnetic Resonance* 285 (2017) 8–17. [PubMed: 29059553]
- [16]. Grant CV, Wu CH, Opella SJ, Probes for high field solid-state NMR of lossy biological samples, *Journal of Magnetic Resonance* 204 (2) (2010) 180–188. [PubMed: 20435493]
- [17]. Odedra S, Wimperis S, Imaging of the B_1 distribution and background signal in a MAS NMR probehead using inhomogeneous B_0 and B_1 fields, *Journal of Magnetic Resonance* 231 (2013) 95–99. [PubMed: 23644349]
- [18]. Nagashima H, Trébosch J, Lafon O, Pourpoint F, Paluch P, Potrzebowski MJ, Amoureux J-P, Imaging the spatial distribution of radiofrequency field, sample and temperature in MAS NMR rotor, *Solid State Nuclear Magnetic Resonance* 87 (2017) 137–142. [PubMed: 28867557]
- [19]. Grant CV, Yang Y, Glibowicka M, Wu CH, Park SH, Deber CM, Opella SJ, A Modified Alderman-Grant Coil makes possible an efficient cross-coil probe for high field solid-state NMR of lossy biological samples, *Journal of Magnetic Resonance* 201 (1) (2009) 87–92. [PubMed: 19733108]

3D-printed forms improve the ease of fabrication for NMR transceiver coils.

Dissolvable inserts are easy to remove without damaging the coil.

This approach can make coil production more reproducible.

Data agreement with simulated magnetic fields demonstrates achievable coil designs.

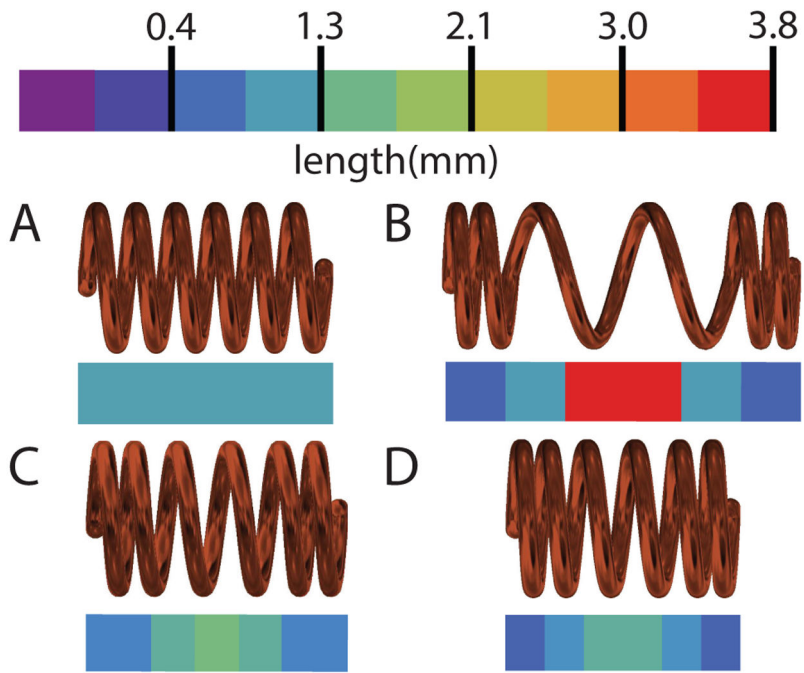


Figure 1:
 The color scale is a graphical representation of pitch. Bars below coils show the magnitude and revolutions of pitch for each design: A) constant, B) stretched, C) model D) ratio.

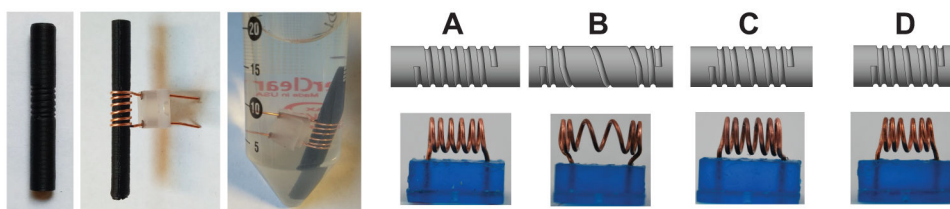


Figure 2:

Shown is a 3D printed DIAPER made from ABS. Copper wire is wrapped around the template to form the coil and positioned in the spinning assembly support. This assembly is soaked in acetone for several hours until the polymer is sufficiently dissolved. CAD designs for the forms and recovered coils are shown from left to right: A) constant, B) stretched, C) model D) ratio. The blue support, which is used for bench testing, is 3D printed using FormLabs Tough V4 stereo-lithography resin.

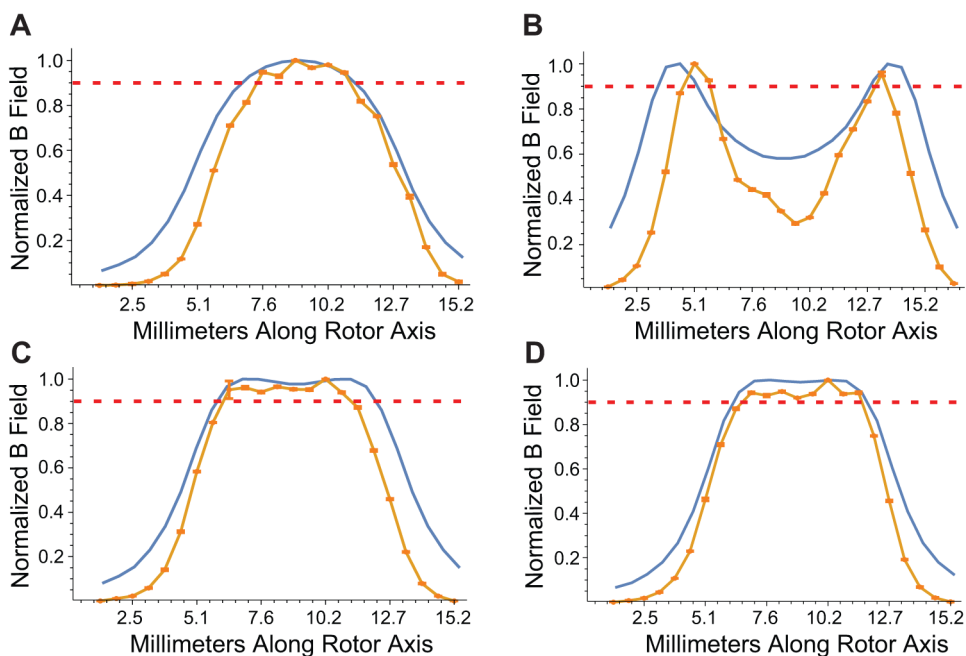


Figure 3:

Experimental and modeled B -field homogeneity for each coil: A) constant, B) stretched, C) model D) ratio. Averaged experimental data performed in triplicate, including error bars, are depicted in orange. The field derived from CST simulation evaluated along the rotor axis is shown in blue. The red dashed line demarcates the acceptable 90% homogeneity threshold. The axial length of the homogeneous region on the constant-pitch design for a 3.2 mm rotor corresponds to viable sample volumes of 11.9 μL simulated and 9.9 μL experimental. Model variable-pitch design resulted in volumes of 17.2 μL simulated and 13.9 μL experimental.

Table 1:
Coil Parameters

Design specifications are listed for each of the four coils in this study. Turns are numbered outside to inside and are symmetric about the center. All pitch dimensions are for one revolution, with the exception of turn 1 pitch on the model coil which is 1.5 revolutions. Inductance was experimentally determined after coil recovery to determine necessary capacitance for simulated tuning in the modeling software.

Coil	Turn 1 Pitch	Turn 2 Pitch	Turn 3 Pitch	Axial Length	Inductance
constant	1.3 mm	1.3 mm	1.3 mm	8.6 mm	72.1 nH
stretched	0.8 mm	1.3 mm	3.8 mm	12.3 mm	66.0 nH
model	1.0 mm	1.5 mm	1.8 mm	8.5 mm	72.5 nH
ratio	0.8 mm	1.1 mm	1.5 mm	7.5 mm	70.3 nH