

Article

Subsurface Spectroscopy in Heterogeneous Materials Using Self-Healing Laser Beams

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Abstract: Self-healing optical beams are a class of propagation modes that can recover their beam shapes after distortion or partial blockage. This self-healing property makes them attractive for use in applications involving turbid media as they can—in theory—penetrate further into these materials than standard Gaussian beams. In this paper, we characterize the propagation of two different self-healing beams (Bessel and Airy) through a solid scattering material with different scatterer concentrations and find that both beams do recover after scattering for samples below a threshold scatterer concentration. Additionally, we test the applicability of both beam shapes for improved sub-surface spectroscopy in heterogeneous materials using fluorescent particles and find that there is an average fluorescence intensity enhancement of $1.3\times$ using self-healing beams versus a standard Gaussian beam.

Keywords: airy; bessel; heterogeneous materials; scattering; spectroscopy; fluorescence; turbid media



Citation: Anderson, B.R.; Gese, N.; Eilers, H. Subsurface Spectroscopy in Heterogeneous Materials Using Self-Healing Laser Beams. *Optics* **2024**, *5*, 310–318. <https://doi.org/10.3390/opt5020022>

Academic Editor: Mario Siciliani de Cumis

Received: 30 April 2024

Revised: 1 June 2024

Accepted: 13 June 2024

Published: 20 June 2024



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1. Introduction

Plastic composites, such as plastic bonded explosives, are heterogeneous materials consisting of various components inside of a polymer host. For instance, PBXs contain high-explosive crystals, grit, plasticizers, antioxidants, and other additives. Given their composition, it is reasonable to assume that the chemistry inside of these materials will be different than at the surface (which is exposed to air). To better understand how the chemistry inside of these materials differs from the surface chemistry, it is therefore necessary to have a diagnostic technique that can see inside of these materials. However, this proves challenging for traditional optical diagnostics (e.g., Raman and fluorescence spectroscopy) as these materials are opaque due to their heterogeneity.

To address this challenge, we recently developed a sub-surface spectroscopy technique [1–4] that utilizes feedback-assisted wavefront shaping (FAWs) [5] to focus probe light inside of a heterogeneous material and onto a guidestar particle; while we have successfully used this technique to probe slow subsurface reactions such as photodegradation [1] and thermal degradation [3,4], this technique is far too slow to probe faster reactions, such as those that occur during detonation [6]. Additionally, this technique requires a guidestar particle to be embedded inside of the heterogeneous material, which can alter the chemistry we are trying to measure.

As an alternative approach (which is faster and does not require a guidestar), we propose to use self-healing laser beams to probe inside of heterogeneous materials. Self-healing laser beams are a class of laser beams (e.g., Bessel, Airy, Pearcey, and Mathieu) that can recover their beam shape after partial obstruction or phase distortion [7]. The self-healing nature of these beams has been used to great advantage to improve propagation through turbulence [8–11], propagation through water [12,13], imaging of biological tissues [14,15], and micromanipulation of small particles/droplets [16–18], and enable new quantum technologies [19–21]. For this study, we rely on the beams' resilience to phase distortion as

our samples are opaque and nonabsorbing. We note that Zhang et al. previously demonstrated a macroscopic analogue of this approach by successfully propagating a Bessel beam through 1 km of turbulent air, while a Gaussian beam produced a speckle-like pattern due to the turbulent air [8].

In this study, we characterize the performance of both Airy and Bessel beams for subsurface spectroscopy using a series of samples containing different concentrations of scatterers. We perform imaging of the self-healing beams transmitted through the scattering samples to determine how resilient they are to scattering and perform fluorescence spectroscopy measurements using Airy, Bessel, and Gaussian beams. Additionally, we consider how the Airy and Bessel beams' shape parameters affect their performance relative to a standard Gaussian beam.

Finally, as an aside, we note that while this is the first demonstration of using any type of structured light beams for subsurface spectroscopy (with other common beam types being Laguerre–Gaussian and Hermite–Gaussian beams) [22–25], there are two other “passive” approaches to subsurface spectroscopy: spatially offset Raman spectroscopy (SORS) and inverse-SORS [26–33]. In both techniques, light is directed onto the surface of a heterogeneous material, while a detector is focused onto a point spatially offset from the beam spot to collect Raman-scattered light. The distance between the source and detection spots are varied, with this distance being correlated with depth based on the scattering properties of the material. Thus, SORS/ISORS can obtain Raman spectra from different depths of the sample. Note that in SORS, the detection spot moves while the light is fixed, whereas for ISORS, the detection spot is fixed and the illumination region is varied. While these techniques can get Raman spectra from different depths of the sample, the resulting signals average over a large area and do not provide localized information, which is the objective of our approach using self-healing beams.

2. Method

2.1. Samples

To demonstrate propagation of self-healing beams through different scattering strengths, we prepared layered polymer samples on 3-inch $\times$ 1-inch glass slides consisting of a bottom layer with 2.5 wt% EYAD in Epotek 305 and a top layer of powdered sugar (0 wt%, 5 wt%, 10 wt%, 25 wt%, and 50 wt%) in Epotek 305 (300 μ m thick). See Figure 1 for the sample structure. We purchased Epotek 305 epoxy from Epoxy Technology (Billerica, MA, USA), Methanol ($\geq 99.8\%$ ACS BDH) from VWR (Radnor, PA, USA), and powdered cane sugar (C&H) from Amazon (Seattle, WA, USA). We prepared the Eu-based complex Eu:Y(acac)₃(DPEPO) (EYAD) as described previously [2]. To prepare the epoxy, we used the manufacturer instructions for a 5:1.4 mix ratio by weight of resin (Part A) to hardener (Part B). After mixing the epoxy, we prepared the bottom layer of the sample (containing epoxy and EYAD fluorescent particles) by mixing EYAD with epoxy and methanol (1:1) with the resulting material spin coated onto a glass slide. To spin coat the bottom layer, we deposited approximately 0.5 g of the thinned mixture onto a glass slide and spin coated at 2500 rpm for 20 s. For preparation of the top layer, we mixed the powdered sugar into the epoxy, with methanol being used to thin the mixture as the concentration of powdered sugar was increased. The weight of the methanol was accounted for when applying each layer. Note that we used vortex mixing throughout to prepare the mixtures for both the bottom and top layers. Once the bottom layer was coated, we first cured it before evenly depositing the top layer. Each layer was cured at 60 $^{\circ}$ C for 1 h. The weight and thickness of each cured spin coated layer was approximately 30 mg and 40 μ m thick, respectively. The cured top layers were approximately 350 mg and 300 μ m thick. We measured the thickness of each layer using calipers and subtracting the thickness of the glass slide.



Figure 1. Layered sample structure used in this study.

2.2. Experimental Setup

Our experimental setup consists of a Verdi V10 Nd:YVO₄ laser (Coherent Corp., Saxonburg, PA, USA) (532 nm, 10 W, <5 MHz linewidth), a Meadowlark SLM (Frederick, CO, USA) (8-bit PCIe controller, 15 μm pixels, 512 $\times$ 512), two long working distance objectives (Mitutoyo M Plan Apo HR 50 $\times$, Aurora, IL, USA, 0.75 NA, 5.2 mm working distance, clear aperture 6 mm), a Point Grey CMOS camera (Richmond, BC, Canada) (GS3-U3-23S6M-C), an Isoplan 320 monochromator (Teledyne Princeton Instruments, Trenton, NJ, USA) ($f = 320$ mm, 600 g/mm, 500 nm blaze) with an attached Blaze CCD (400-B, 1340 $\times$ 400, 20 μm), and various optics (e.g., lenses, mirrors, polarizers, half-waveplates). See Figure 2 for a schematic drawing of the experimental setup.

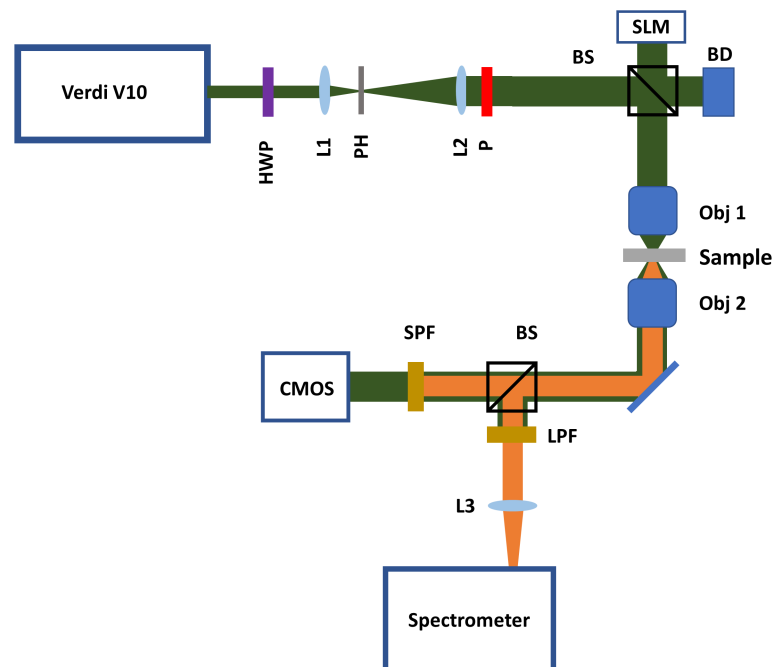


Figure 2. Schematic of experimental setup used to test self-healing beam propagation and spectroscopy. HWP: halfwave plate; L1/L2/L3: lenses; PH: pinhole; P: polarizer; BS: beam splitter; BD: beam dump; Obj 1/2: objectives; SPF: short-pass filter; LPF: long-pass filter.

To produce the Airy and Bessel beams, we illuminated the SLM with a TEM₀₀ Gaussian beam and set the SLM phase mask to [34]

$$\Psi = \frac{1}{a^3} (k_x^3 + k_y^3), \quad (1)$$

for an Airy beam—where a is a scale parameter, k_x and k_y are the spatial coordinates of the SLM pixels (with the origin at the center of the SLM)—and [35]

$$\Psi_n(k_\rho, \phi) = n\phi + \frac{2\pi}{b} k_\rho, \quad (2)$$

for a Bessel beam of order n , where b is a scale parameter, and (k_ρ, ϕ) are cylindrical coordinates on the SLM with the origin at the center of the SLM. Note that in this study, we will only be considering zero-order Bessel beams which have a central bright spot and rings of decreasing intensity. These differ from higher-order Bessel modes which have a central dark spot. Figure 3 shows an example phase mask that was used and their resulting experimental beam profiles.

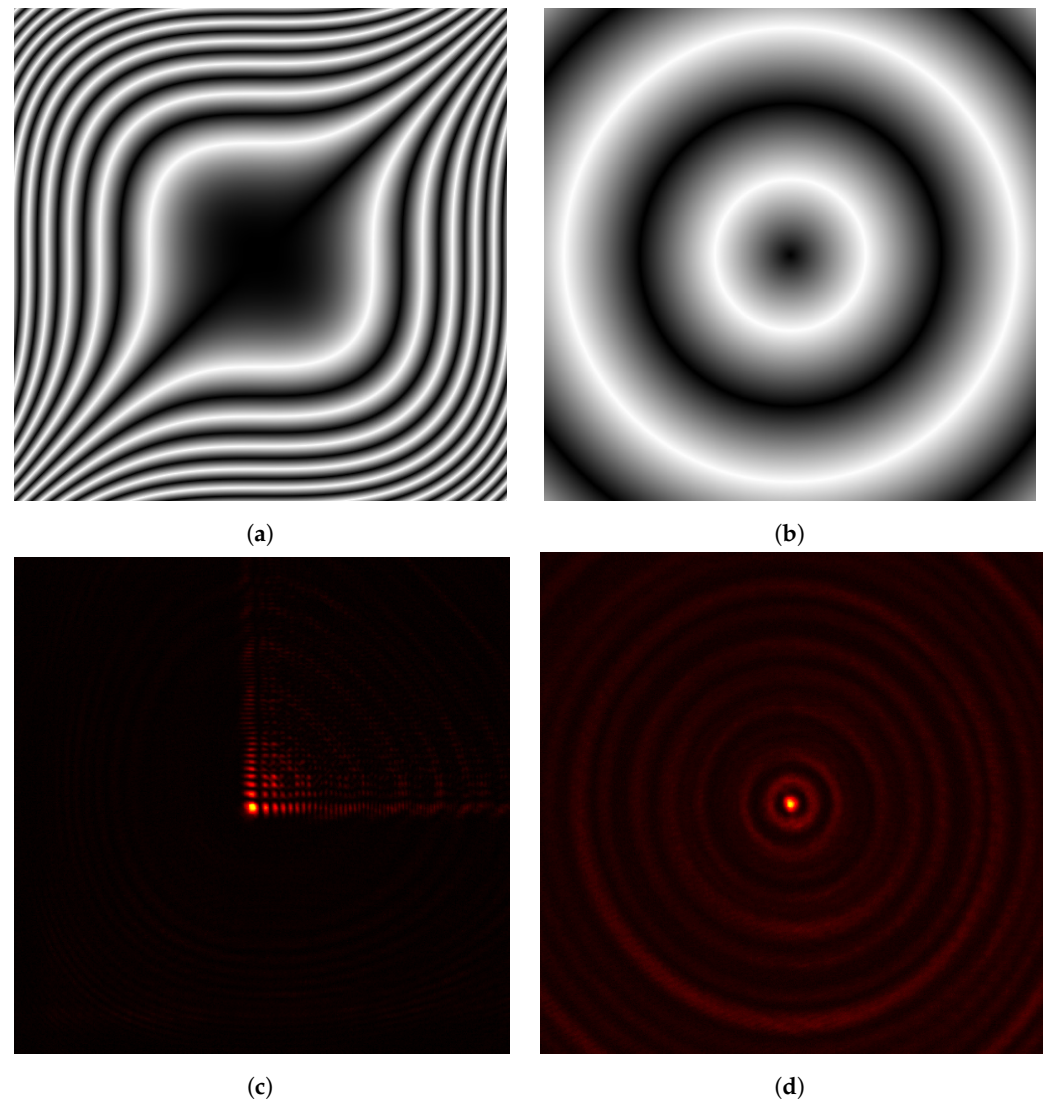


Figure 3. Example Airy and Bessel phase masks (a,b) and their corresponding beam profiles (c,d). Note that for the phase masks the Airy shape parameter is $a = 100$ and the Bessel shape parameter is $b = 60$.

3. Results and Discussion

From Equations (1) and (2), we see that the both the Airy and Bessel phase profiles have a single adjustable parameter that affects the resulting beam shape. Additionally, we note that the beam shape at the focus of the microscope objective is also affected by the objectives focal length. However, this is a fixed quantity for our experiments and cannot be varied. To determine the optimal beam parameters for the Airy and Bessel beams, we measured the peak fluorescence intensity from an EYAD particle inside a 50 wt% sample using a series of phase masks generated from Equations (1) and (2) using a range of beam parameters. Additionally, we measured the peak fluorescence intensity for a Gaussian beam for reference. Figure 4 shows the resulting peak intensities for the Airy and Bessel beams for a range of beam parameters. From Figure 4, we find that the measured fluorescence

intensity depends strongly on the beam parameter, with only certain beam parameters resulting in intensities higher than the normal Gaussian beam. Namely, the Airy beam peaks at $a = 100$, while the Bessel beam peaks at $b = 60$. Note that these peak locations are consistent across the different sugar concentrations but depend on the choice of objective focal length as the Airy/Bessel beam quality depends on both the beam parameter and focal length.

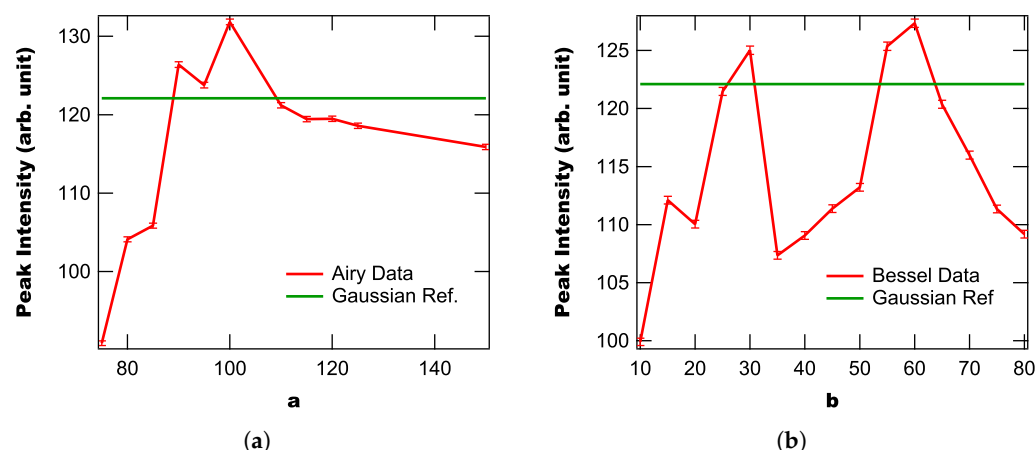


Figure 4. Peak fluorescence intensity as a function of beam parameter for an Airy (a) and Bessel (b) beam using a 50 wt% sugar concentration sample.

Having determined the optimal beam parameters for our Airy and Bessel beams, we next performed a series of fluorescence measurements using all five samples and all three beam types. For these measurements, we located five separate EYAD particles in each sample and illuminated the samples with each of the three beams while measuring the resulting fluorescence spectra. Figure 5a shows example spectra measured from one spot on the 25 wt% sample using each beam type, with both the Airy and Bessel beams producing greater fluorescence intensities. Note that these intensities are scaled to the beam power incident on the sample to account for variations in pump power due to beam shaping.

After measuring the power-scaled peak intensities at five points on each sample, we next computed the average intensity ratios between the Airy/Bessel beams and the Gaussian beam for each sample, with Figure 5b showing the computed ratios and their standard deviations for each concentration. From Figure 5b, we find that for all sugar concentrations (including no sugar) the self-healing beams produce greater fluorescence intensities than a standard Gaussian beam, with the largest improvement being $\approx 1.55\times$ for the Airy beam and the 0 wt% sample and the average improvement being $1.3\times$. Note that in our previous work using FAWS, we achieved enhancements of up to $40\times$.

The fluorescence intensity enhancement when using the Airy/Bessel beams can be attributed to the Airy/Bessel beams' resilience to phase distortions due to the self-healing effect. When each beam is incident on the sample, there is both phase distortion at the polymer surface (which is why we observe enhancement in the 0 wt% case) and scattering from the embedded sugar particles. The self-healing beams' resilience to this distortion/scattering results in the beams maintaining their compact form, while the Gaussian beam diverges due to the distortion/scattering. To further demonstrate this difference, we plot example fluorescence images in Figure 6 for each beam and the 50 wt% sample with several fluorescent particles nearby to each other. For the Gaussian beam, the excitation is broad and excites all four particles. However, in the case of the Airy beam only, the primary particle and the nearest particle are excited, while for the Bessel beam, there is some excitation of the farther particles but less than observed for the Gaussian beam. These images show that the excitation beam profile at the target particle is more compact for the Airy/Bessel beams than for the Gaussian beam, which we attribute to the Airy/Bessel beams' resilience to phase distortion.

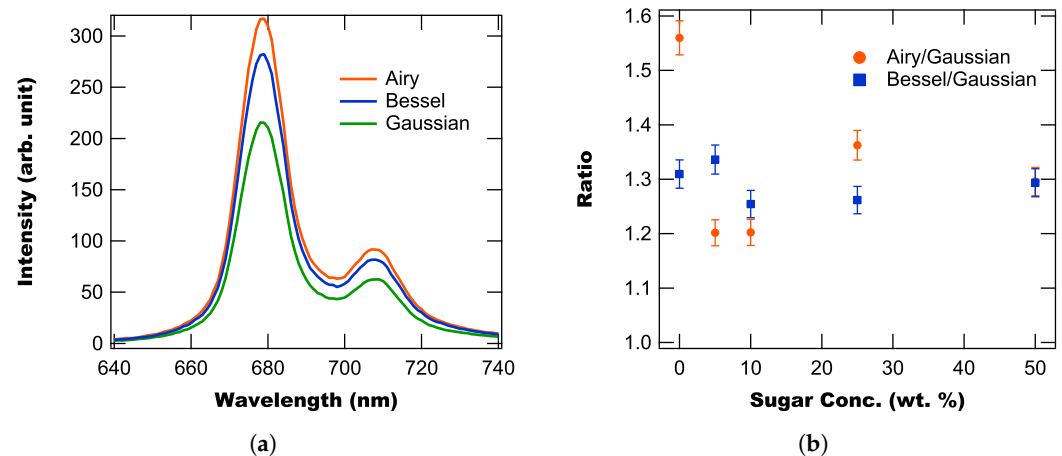


Figure 5. Example fluorescence spectra measured from the 25 wt% concentration sample for the three beam types (Airy, Bessel, and Gaussian) (a) and the sample-averaged intensity ratios as a function of sugar concentration (b).

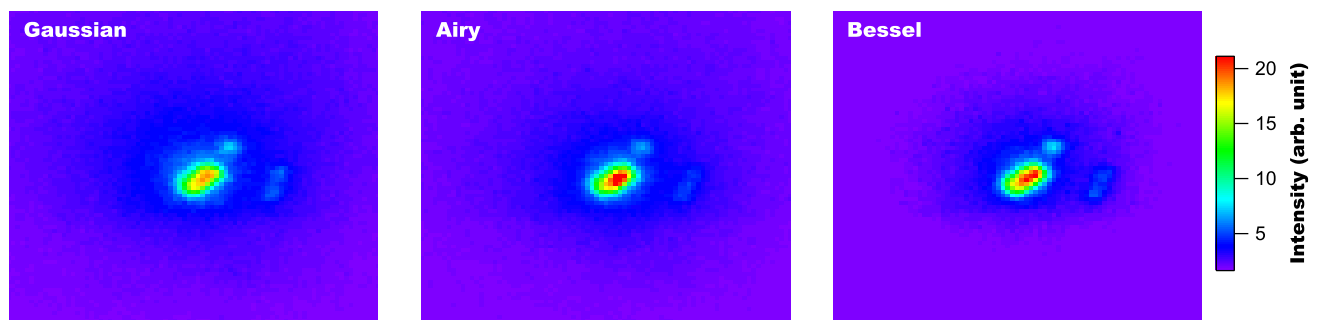


Figure 6. Fluorescence images of EYAD particles for the three different beam types for the 50 wt% sample.

To better understand how each beam type behaves when transmitted through the scattering layer, we imaged each beam type transmitted through the scattering layer for each sugar concentration, with the resulting images shown in Figure 7. From Figure 7, we find that for the Gaussian beam, a speckle pattern emerges as soon as we introduce scattering with the speckle grains decreasing in size as the concentration increases. However, when we consider the self-healing Airy and Bessel beams, we find that the beam shape is partially recovered after passing through both the 5 wt% and 10 wt% concentration samples, while a speckle pattern is observed once again for concentration of 25 wt% and 50 wt%.

Additionally, we take line profiles across the center of the beams to better visualize the shape of the intensity patterns, with Figure 8 showing the normalized line profiles for each beam and sugar concentration. From Figure 8, we make several observations. First, we find that for the 0 wt% sample the beam shape is roughly Gaussian with fluctuations, which we attribute to phase distortion caused by the polymer's surface not being perfectly flat. However, both the Airy and Bessel beams display expected profiles, which we attribute to their resistance to distortion. Next, we find that as the sugar concentration increases to 5 wt% and 10 wt% the Gaussian beam's transmitted line profile drastically diverges from the 0 wt% beam shape, while both the Airy and Bessel beams are only slightly modified. Finally, for the 25 wt% and 50 wt% line profiles, we observe that while there is speckle in all three beams, the Airy beam still maintains some similarity to the original beam with the speckle being more localized.

Thus far, we have been considering a qualitative comparison of the different beam shapes after passing through the sample. However, we can also perform a quantitative comparison by calculating the Pearson's correlation coefficient between the different sugar concentrations and the 0 wt% image. Figure 9 shows the resulting correlation coefficients

as a function of sugar concentration. From Figure 9, we find that as the sugar concentration increases, the correlation coefficient decreases for each beam type, but for all concentrations, the Airy and Bessel beam outperform the Gaussian beam. This is expected as both the Airy and Bessel beams are more resilient to phase distortion.

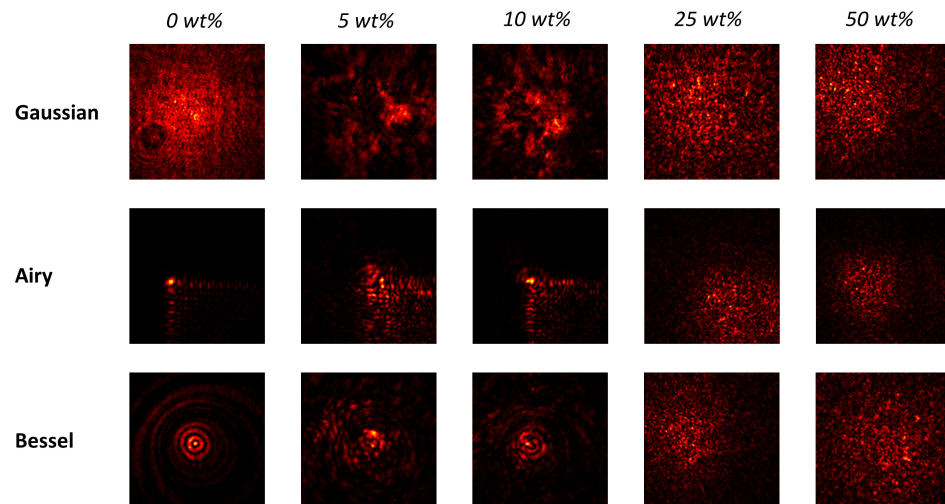


Figure 7. Example images of beams transmitted through heterogeneous samples containing different sugar concentrations for Gaussian, Airy, and Bessel beams.

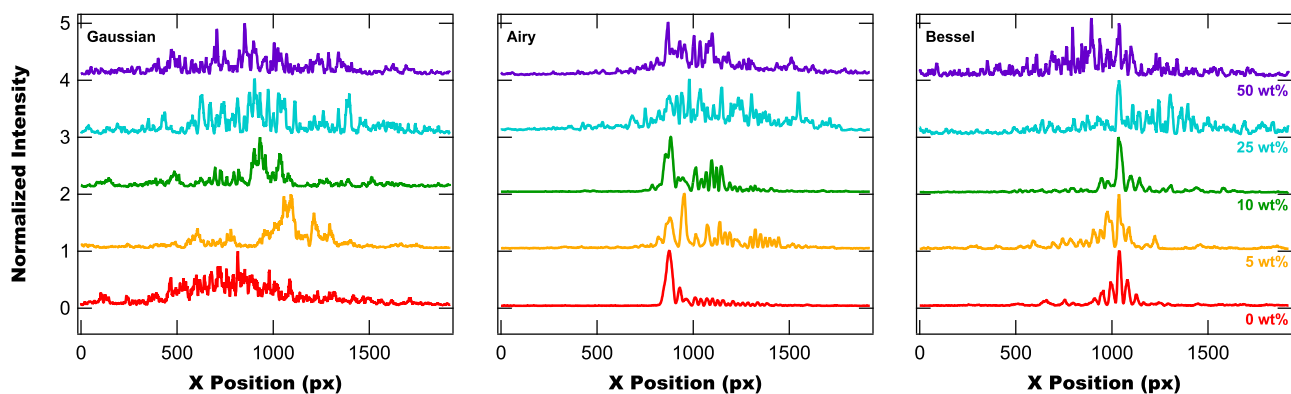


Figure 8. Line profiles across transmitted beam images for each beam type and concentration.

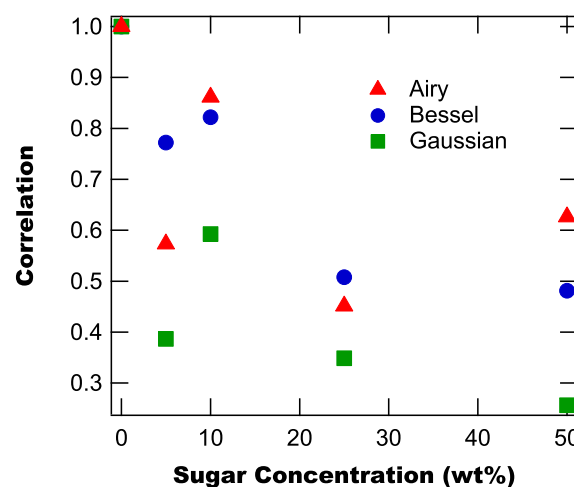


Figure 9. Correlation coefficient between 0 wt% beam and different sugar concentrations for each beam type.

4. Conclusions

Self-healing laser beams are a useful class of beam shapes that can aid in propagation through turbid media due to their ability to reform their shape after an obstruction or distortion. In this study, we have tested two different self-healing laser beam shapes (Airy and Bessel) for propagation through heterogeneous polymer samples at different scatterer concentrations. We find that for the samples used, the self-healing beams are capable of recovering their beam shapes after samples containing 5 wt% and 10 wt%, but higher concentrations result in disordered speckle patterns.

We also test the feasibility of using these beams for subsurface spectroscopy inside heterogeneous materials by preparing samples containing fluorescent EYAD particles on a glass slide coated by a heterogeneous polymer matrix containing sugar crystals. We find that for all scatterer concentrations, the Airy and Bessel beams provide improved spectral intensity as compared to a standard Gaussian beam with the mechanism of this improvement being a better resilience to phase distortion. However, while we observe improved intensity with the Airy and Bessel beams, we find that the observed enhancement is $<1.55\times$ with an average value of $1.3\times$ which is negligible compared to other techniques that can obtain enhancements of $40\times$ or greater. These results imply that while this approach has promise at propagating these beams through scattering materials (with sufficiently low scatterer concentrations, it is not a viable technique for improving subsurface spectroscopy in heterogeneous materials.

Author Contributions: H.E. and B.R.A. conceived of the experiments. N.G. prepared the samples. B.R.A. performed the experiments, data analysis, and wrote the manuscript with input from all authors. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Nuclear Security Administration, Award # DE-NA0004083 to Washington State University.

Data Availability Statement: Data underlying the results presented in this paper are available from the authors upon reasonable request.

Conflicts of Interest: The authors declare no conflicts of interest.

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