

Experimental Validation of the Dynamic Molecular State of Water in Damaged Polymer Composites Using Near Infrared Spectroscopy

OLUWATIMILEHIN OLUWAJIRE, KATHERINE BERKOWITZ,
RISHABH D. GUHA and LANDON GRACE

ABSTRACT

Safety has long been a key factor in the design, manufacturing and maintenance of products that are made from composite materials. The exceptional properties these materials exhibit compared to their metal counterparts is enabling widespread adoption across civil infrastructure, oil & gas, marine, automotive, and aerospace industries. But the lack of a definitive and accurate technique to predict damage progression in a polymer-matrix composite (PMC) during their service life continues to pose a major risk and creates a gap in the long-term integrity of the structures produced. Although there is widespread consensus regarding the deleterious effects of the ingressed moisture on the overall properties of a composite, recent studies have revealed that the inevitable presence of moisture in a PMC can be leveraged for damage characterization. This work aims to employ Near-Infrared spectroscopy for quantifying molecular moisture in polymer composites for sub-micron scale damage detection.

Prior to moisture absorption, a drop tower was used to induce a barely visible impact damage (BVID) in the center of dry E-glass/epoxy specimens. Three different specimens were subjected to 1J, 1.5J, and 2J of damage, respectively. The NIR Nano EVM Spectrophotometer was used to obtain spectral scans between wavelengths of 900-1700 nm for each of the three damaged samples, as well as an undamaged sample, in their dry state. The samples were then exposed to moisture contamination via water bath, and subsequent spectral scans were acquired at consistent intervals of gravimetric moisture gain. The spatial variation of the moisture content was evaluated from the characteristic peak for water in the damaged samples at various levels of absorbed moisture. The absorbance area obtained from the NIR spectral shows quantitative values to represent increasing damage and spatial maps indicating different states of absorbed moisture in each sample.

North Carolina State University, 1840 Entrepreneur Dr, Raleigh, North Carolina 27695, U.S.A.

INTRODUCTION

The ever growing need to optimize existing materials, develop new ones, and bring to fruition complex designs afforded by our unbounded imagination cannot be overemphasized. The inherent challenge, however, is not just limited to manufacturing these complicated engineering parts but also safety concerns and considerations of these products [1]. Advanced composites have emerged as valuable material systems that can be used to achieve multifaceted designs; maximizing their anisotropic properties and utilizing their high specific properties improves material functionally in applications where traditional metals and other polymers have limitations [2]. Even though composites have long been around and have seen growing application in aerospace, marine and construction industries, they continue to drive the development of new multifunctional products in industries like infrastructure, transportation, sports and recreation. [3-5]. However, their widespread use and complexity raises the need for non-destructive damage evaluation [6]. Unlike metals, polymers and other engineering materials, composites tend to have complex failure mechanisms which require accurate and dependable methods of evaluation to ascertain their long-term structural reliability [7].

Destructive and non-destructive evaluation (NDE) methods used in detecting and predicting flaws in metal structures have gone through a lot of developments and are in a mature stage. Conversely, NDE methods of evaluating damage in composites are still in their nascent stages and have not been investigated enough to give accurate evaluation of advanced composite materials [7,8]. Polymers and polymer composites have been evaluated using established NDE procedures which includes visual inspection, ultrasound, fiber-optic laser-ultrasound, infrared thermography, x-ray radiography, x-ray computed tomography, acoustic emission, and shearography, among others [9-14]. These methods have only been moderately successful in assessing damage in polymer composites despite their widespread use. There is a need to develop innovative approaches and validate existing ones to accurately detect and better quantify damages below the micro-scale [15].

Idolor et al previously used the dielectric cavity method, also known as the split post dielectric resonator (SPDR), to assess and locate damage in a glass fiber polymer composite laminate via the differences in relative permittivity of absorbed moisture [8]. The certainty of moisture absorption throughout the lifetime of a composite material and deleterious effects it has on such samples has been thoroughly explored in literature [22,25-26]. Making use of polymer-water interaction and damage dependent hysteresis, an opportunity for damage detection in polymer matrix composites was investigated—leading to the discovery of moisture dependence criteria as a basis of nondestructive damage detection [15]. However, the restrictions and limitations of this method in terms of space, thickness of sample and scan time makes it apparent that there is the need for a more efficient method that would be quick, non-destructive and provide a quantitative parameter to effectively characterize damage in polymer composites.

Presented here is an effective alternative way of analyzing damage in polymer composites using the Near Infrared Spectroscopy (NIR) technique combined with the established idea of polymer-water interaction [15]. Because of its capacity to offer quantitative information on a variety of products quickly, NIR analysis has become increasingly popular [16]. NIR has vastly found implementation in food science,

pharmaceutical industry, agriculture and as a non-invasive technology in clinical chemistry [17,18]. The deployment of this technology for detecting and quantifying damage in composite materials has the potential to be a viable non-destructive technique for in-service advanced composites. NIR can identify damage in a polymer composite by analyzing the distribution and state of absorbed water molecules. Water interacts with the polymer network in various ways, from firmly bound direct hydrogen bonds to less restrictive interactions such as van der Waals forces [19]. The state of water within the polymer network is dependent on the physical and chemical state of the polymer itself. Although little has been done to study the molecular state of moisture absorbed during the service life of a composite using the Near Infrared Spectroscopy (NIRS), it has the potential to offer a great insight into damage detection and monitoring. This could be used to develop a NDE technique that will transform damage monitoring in polymer composites.

This article corroborates the idea of using the molecular state of moisture absorbed in composite materials as an “imaging agent” to characterize damage progression at various levels of absorbed moisture. The absorbance area obtained from NIR Nano EVM Spectroscopy Spectra analysis was used as a quantitative parameter to represent the molecular states of total moisture at distinct levels of damage.

MATERIALS AND METHODS

Material Manufacture

Four test samples were manufactured from an epoxy/glass fiber laminate typical of the aerospace industry. A 12-inch x 12-inch laminate was formed from 10 plies of prepreg while the orientation of 0° and 90° directions was maintained. The prepreg with the trade name Hexcel F-161 consists of an epoxy resin which has the glass fiber reinforcement of 8-harness satin weave glass fabric, style 7781. In accordance with the manufacturer specifications, a hot press kept at 50 psi was used for the curing process with the temperature raised from room temperature to 350°F. The sample laminate was held at this temperature via the hot press for 2 hours before the pressure was released. After this, the sample was allowed to cool freely to the ambient temperature of the lab (~70°F). The test samples were cut from the manufactured laminate with a waterjet. Each sample has a 130mm x 75 mm dimension with an average thickness of 2 mm.

Specimen Preparation and Impact Setup

A vacuum oven was used to dry the rectangular cut specimens at 65°C until a stable weight was achieved in accordance with the ASTM D5229 specifications [20]. Each sample was subjected to one low-velocity impact event using a drop tower. A crosshead with a semi-hemispherical striker tip attached to a double column impactor guide mechanism was dropped from the appropriate heights to induce a central impact on each of the samples. This created a barely visible impact damage (BVID) like what might be seen from hail impact in-service. Three samples were impacted using energies of 1, 1.5, and 2J, respectively.

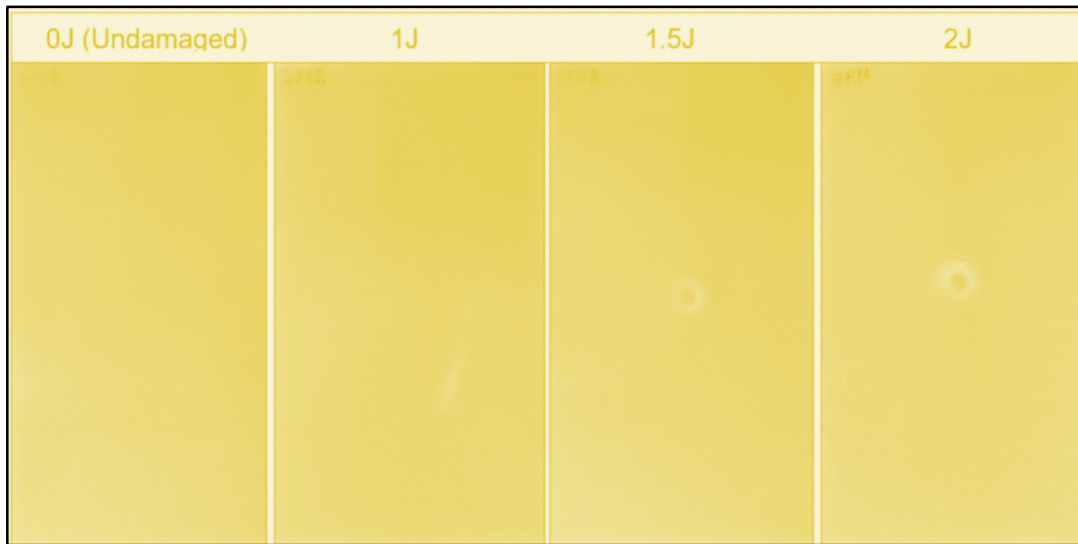


Figure 1. Test samples L-R with 0J, 1J, 1.5J and 2J damage.

Moisture Contamination

Before introducing moisture, the measurement of the weight of each sample was taken using a high precision analytical balance. Near Infrared data of the dried samples were obtained and recorded prior to the start of immersion in moisture.

The samples were immersed in a constant temperature water bath containing deionized water maintained at 25°C. Near Infrared absorbance readings were collected for each sample at intervals of every 0.05% increase in gravimetric moisture content. To stabilize and control the readings at each moisture content, the sample was dried with a lint-free cloth and kept at ambient temperature for 20 minutes. This helps to eliminate the effects of surface moisture as well as reduce the rate at which moisture is lost after being removed from the water bath thereby minimizing the uncertainties across measurements. Static electric charges on the specimen surface were removed just prior to weighing by exposing it to air containing positively and negatively charged ions supplied by an ionizing gun.

Near-Infrared setup

The NIR absorbance spectra was obtained from NIR Nano EVM Spectrophotometer with wavelengths of spectral scans between 900-1700 nm. To obtain accurate NIR scans across the four samples, a setup of NEMA-17 stepper motors, linear screw actuators, and A4988 motor drivers are used as shown in Figure 3. Movement of the screw actuators were done manually and a set of grids with 12 points across the length of the sample was accurately positioned to obtain the measurements as shown in Figure 3. A step size of 1 cm was used to maximize the possible mapping space across the length of the sample.

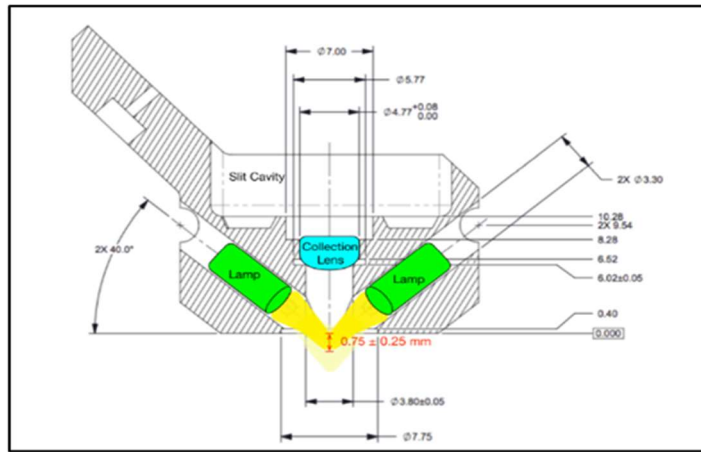


Figure 2. NIR Illumination module.

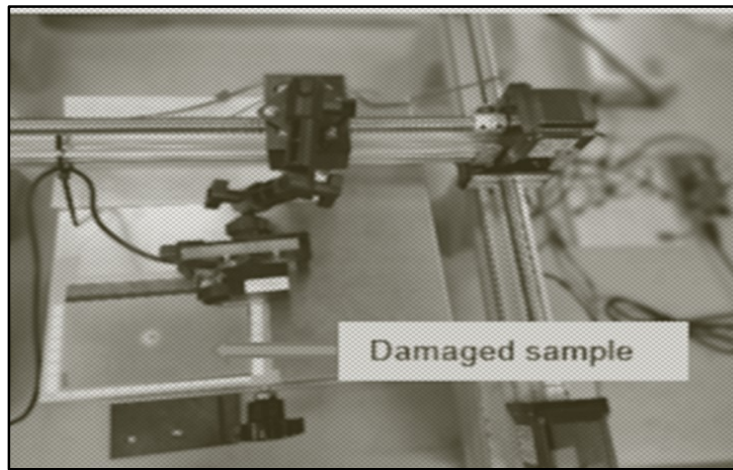


Figure 3. Scan experimental setup.

Near-Infrared Spectroscopy

The sample absorbs a set amount of NIR light during a scan and diffusely reflects the non-absorbed light into the system. The amount of light absorbed at each wavelength is determined by the substance's molecular makeup and is unique to that material, giving it a chemical fingerprint. The illumination module shown on Figure 2 above has a collecting lens that collects diffusely reflected light from the sample and focuses it into the optical engine through the input slit. The slit size is set to balance wavelength resolution with the spectrometer's Signal Noise Ratio (SNR). This spectrometer uses a $25 \mu\text{m}$ wide by 1.8 mm tall slit. The light that passes through the slit is collimated by the first set of lenses, passes through an 885 nm long wave pass filter, and then strikes a reflective grating. This grating, in combination with the focusing lens, disperses the light into its constituent wavelengths. The optical system images 900-nm wavelengths to one end of the Digital Micromirror Device (DMD) and 1700-nm to the other end, with all other wavelengths sequentially dispersed in between [21].

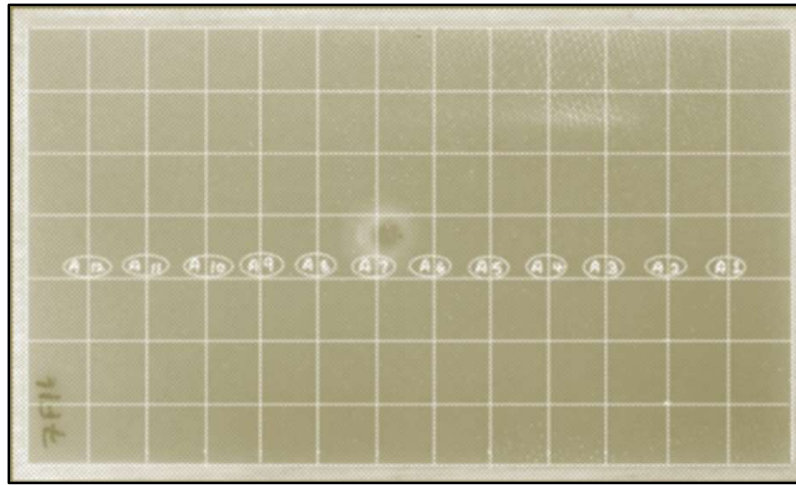


Figure 4. Twelve points grid division showing scan area of a sample.

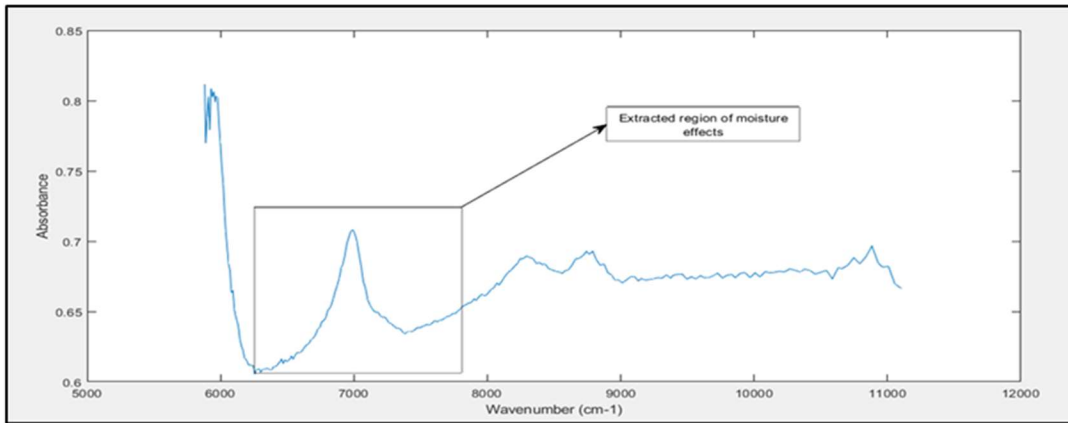


Figure 5. Typical NIR spectra for moisture contaminated epoxy highlighting extracted region of moisture effect.

Figure 4 shows the grid of a sample with points A1 to A12 where NIR scans are taken across the center of the sample. As an output from the NIR scan, the Absorbance vs Wavenumber spectra is obtained. Figure 5 shows the entire NIR spectra recorded for a single point from which the appropriate frequency range (between 6000 and 8000 cm^{-1} , where relevant water behavior can be monitored in the spectra) was extracted [22]. The data was analyzed in three steps: baseline subtraction, initial dry spectra subtraction, and calculating the absorbance area values from the obtained spectra [1]. The spectral data was subjected to an objective baseline correction technique based on prior study [23]. This entailed fitting the data with an n-degree polynomial and then removing points that were greater than one standard deviation above the fitted line. This procedure was repeated until the curve converged to the peak's bottom, forming the requisite baseline. According to preliminary observations, a linear baseline might be appropriate for this spectral range [23]. This approach was used to fit a linear function to points on both ends of the relevant spectral range in all circumstances, and then subtract the resulting linear baseline from the spectra.

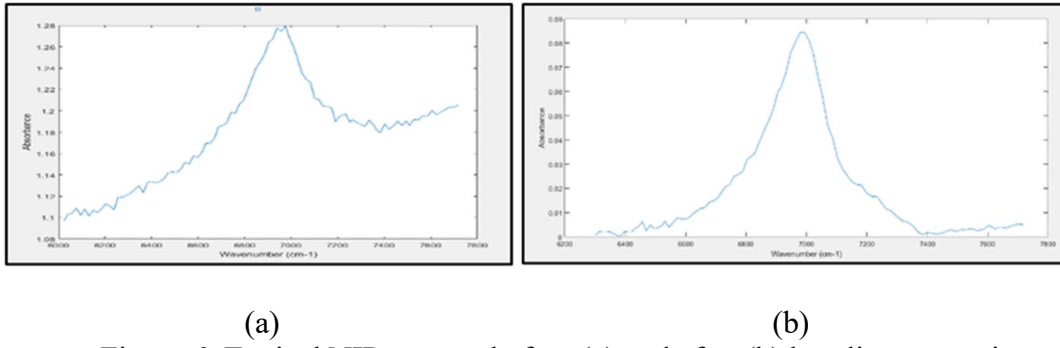


Figure 6. Typical NIR spectra before (a) and after (b) baseline correction.

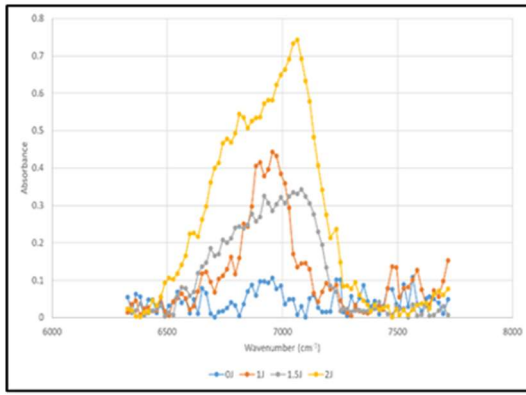
To reduce spectral contributions from epoxy and glass fiber constituents of the polymer matrix composite, the baseline-corrected dry spectra were subsequently removed from all subsequent baseline-corrected moisture-contaminated spectral conditions [22]. Figure 6a and 6b shows a spectra curve for a damaged sample with moisture contamination before and after baseline correction and dry spectral subtraction.

RESULTS AND DISCUSSIONS

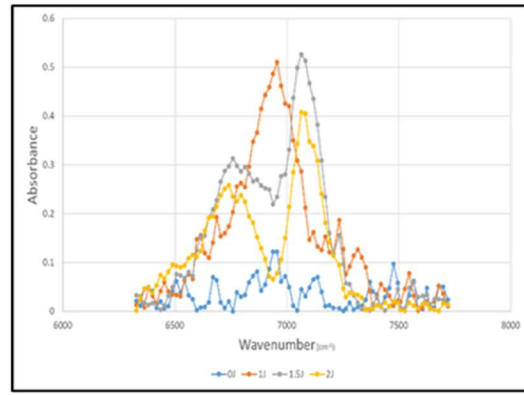
The NIR Spectroscopy is sensitive to moisture content, amount, and type of damage as well as the material/chemical constituents of each sample. We have focused on spectral data obtained in the region between 6000 cm^{-1} to 8000 cm^{-1} where water can be separated into its dual (free and bound) states [19,22]. Thereby, considering the absorbance values primarily due to damage and moisture effects as a means of detecting and quantifying damage in these samples. Previous studies have shown the effects of specimen thickness variation were reduced by obtaining data from dry, undamaged samples before introducing damage to each sample. Therefore, absorbance values obtained for each scan post-moisture contamination were adjusted by subtracting the corresponding dry baseline scan. For each scan, a spectral of absorbance values within the specified wavenumber is obtained. The absorbance area for each point was calculated as the sum of the area under each spectral curve using equation:

$$A = \sum_{i=1}^n (a1 + a2) * (w2 - w1) \quad (1)$$

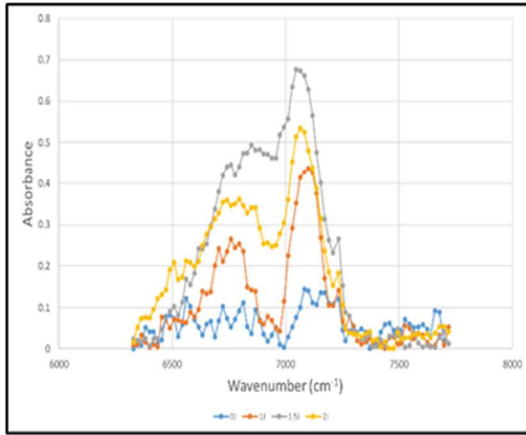
Figure 7a-d shows spectral curves of absorbance area against the wavenumber (cm^{-1}) for the center point in each sample where the impactor was dropped on each sample, causing an impact damage. This point corresponds to the line labeled A7 shown in Figure 3 above and is the area where the presence of moisture is prevalent compared to other points in the sample. With increasing levels of damage, 0J (no damage), 1J, 1.5J and 2J, a corresponding increase in the absorbance peaks was observed. From the plots, it is noted that at lower moisture contents of 0.05% and 0.01% there is less distinction in the peaks of the absorbance spectra compared to higher moisture contents of 0.15% and 0.2%. This is because absorbance values vary more significantly from that of dry samples at higher moisture contents.



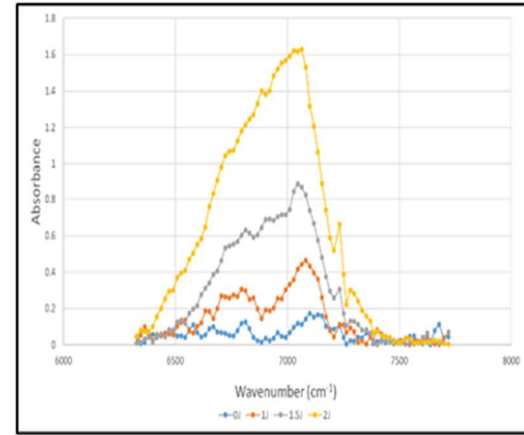
(a)



(b)



(c)

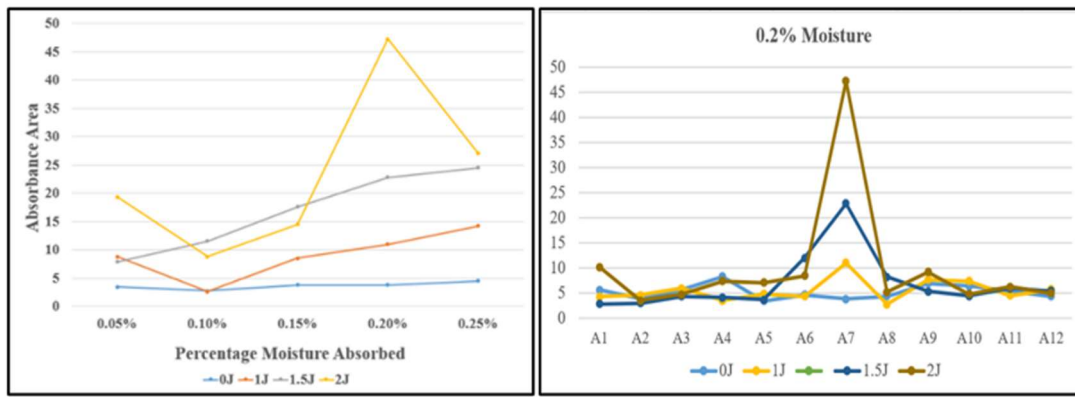


(d)

Figure 7. Absorbance plots at (a) 0.05, (b) 0.10, (c) 0.15 and (d) 0.20 percent moisture content for increasing levels of damage.

Higher absorbance peak of the 1.5J sample compared to the 2J peak at 0.10% moisture might be due to sampling errors at the point of manual data collection. To mitigate this, work is ongoing to use an Arduino board control coupled with MATLAB scripts to sequentially control the position of the NIR setup.

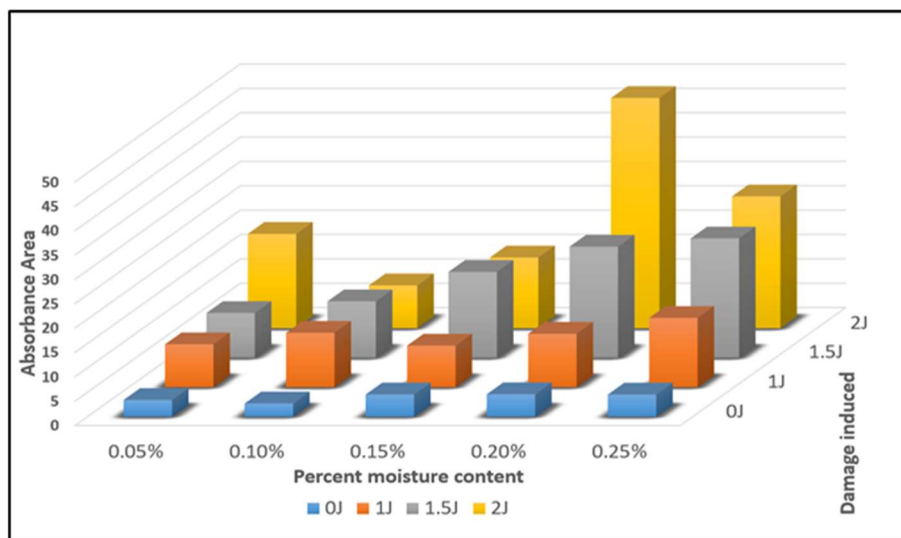
To better understand the differences between each sample and quantitatively classify each sample, we obtained the total area under the spectral curves which represents the amount of moisture present at that specific point in the sample.



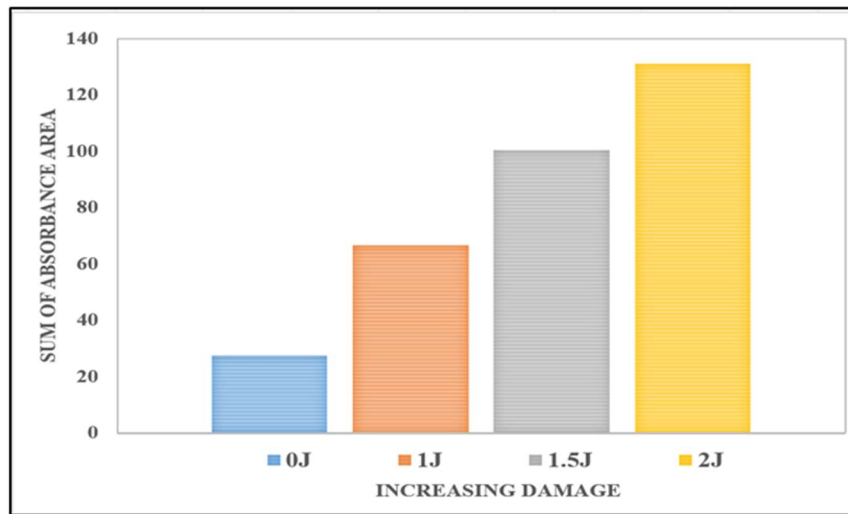
(a) (b)

Figure 8. Absorbance area for increasing moisture content for distinct levels of damage.

Figure 8a shows the variation of absorbance area with increasing moisture at point A7 - the midpoint of damage as captured by the NIR. While Figure 8b shows the same variation at 12 points along the center of a sample (Fig. 4) at 0.2% moisture content for increasing damage. Overall, there is an increase in the absorbance values for increasing percentage of moisture. The peak at the damaged area, point A7, becomes more and more pronounced with increased damage content, indicating that at higher levels of damage there is more moisture occupying the damage site. The sum of all the absorbance area for points A1 to A12 on each sample is shown in Figure 9a for increasing moisture content and increasing damage while Figure 9b shows the sum of all the absorbance area at all the moisture contents considered up till 0.25%.



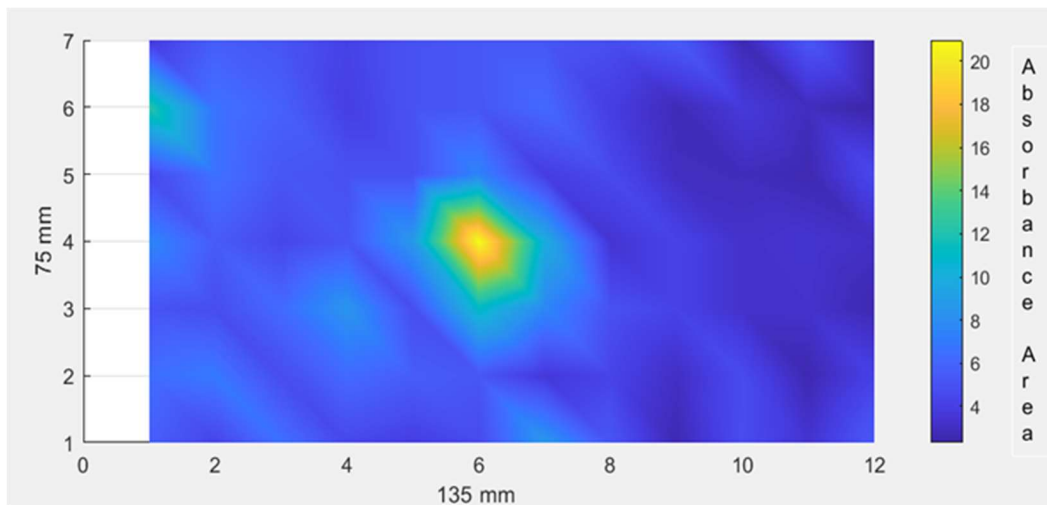
(a)



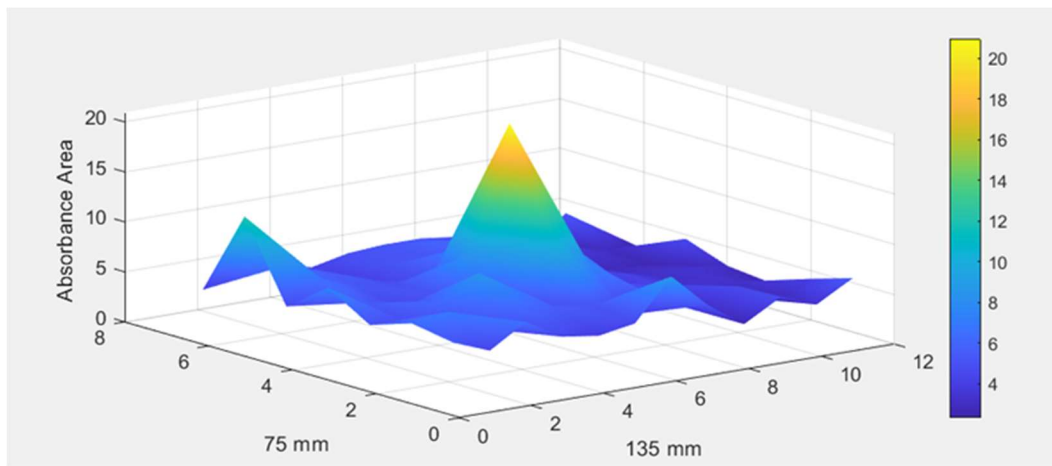
(b)

Figure 9. Comparison of absorbance area under curve with increasing moisture content and levels of damage.

As observed in Figure 9a, the absorbance area values are not increasing in an exactly linear manner as ideal for increasing moisture content and increasing damage. The variability seen is perceived to occur because of an opposing effect between the moisture content contribution and the damage contribution as detected by the NIR Spectroscopy. While the moisture present contributes to the increase in absorbance area, the increased amount of damage reduces the absorbance area. In an endeavor to better understand the distribution of absorbance area at multiple points throughout the entire sample, a scan of eighty-four (84) points was done using the NIRS. 2D and 3D area maps were obtained as shown in Figure 10a and 10b for a 1.5J damage sample at 0.20% moisture content.



(a)



(b)

Figure 10. Absorbance Area maps of 1.5 J damage sample: (a) 2D map (b)3D map.

The area maps reflect the distribution of moisture in the damaged sample based on the absorbance area obtained from the NIRS grid scan depicted in Figure 4. From the figure we can infer that the absorbance area values remain close at points where there is “no damage” (depicted with blue coloration in the map) and significantly higher at points where the impact damage is higher (depicted in the map as yellow gradient). Previous studies highlight that moisture absorbed can be classified into its free and bound states in a composite sample [22]. The absorbance areas obtained from the spectral scans and plotted as area maps in Figure 10 supports the distribution of moisture in the damaged sample into the free and bound states. Sites with more damage (toward the center of the sample) have higher absorbance area values while further away from the center of the sample have lower values of absorbance area indicating the presence of moisture. This suggests that regions of higher absorbance represent areas where more free water exists, and lower absorbance areas could represent areas where water exists in its bound state within the sample. This supports the hypothesis of the dual state of moisture in a damaged composite material and can be used to detect previously unknown damage.

Some points away from the exact spot of damage have higher absorbance values, indicating potential damage in forms of microcracks, delamination or other forms of defects that might have been propagated into the sample due to the impact at the center of damage. It also could indicate voids or defects formed during manufacturing. These forms of damage, though not seen by the naked eye, may be detectable using the NIR Spectroscopy technique. This, however, will require validation using another technique such as Atomic Force Microscopy (AFM). An advantage of this technique is the ability to evaluate parts which are accessible from one side only. These types of parts are typical for in-service composite applications.

The most crucial step in using the NIRS is a thorough consideration of experimental design. Once established, one can enjoy the benefit of speed and avoiding mistakes, an advantage that is valuable to engineers [17]. However, the NIR approach may meet limitations for materials that have a high absorbing potential since the absorbance area depends on the ability of the sample to reflect infrared light.

Also, the data analysis of the NIR has been noted to be challenging [17]. Potential sources of errors could be from actual NIR spectral scans which might not be taken at the same environmental conditions. The effects of ambient temperature, humidity and other human effects during the scans could also lead to errors which can be minimized by automating the scanning and analysis process. The hypothesis is that using this absorbance area data, we can predict the level of damage not previously known in a sample. In actual practice, the amount of damage in a sample (if any) would be the needed parameter to be known which in turn can be used to predict the failure progression of the material over a period. This method, when fully developed, has the potential to help detect and quantify damage at the micron level which will prevent catastrophic failure in advanced composite materials.

CONCLUSION

This study quantified damage in composites that may or may not be visible to the naked eye by leveraging the tendency of composites to absorb moisture. Near Infrared Spectroscopy (NIRS) scanner was used to obtain spectral data about the moisture content within four test samples manufactured from an epoxy/glass fiber laminate typical of the aerospace industry. As an output from the NIR scan, the Absorbance vs Wavenumber spectra was obtained. Results obtained from the spectral data shows spectral curves of areas under moisture influence with increasing degrees of moisture contamination for varying levels of damage in the samples. By quantifying the amount of damage using the absorbance area data, it is observed that for each level of moisture, there is an increased amount of water absorbed with increasing amounts of damage on each sample. This supports the premise that moisture effects can be used to detect damage and its progression in composite samples [15,22]. The study further demonstrates that the absorbance area values obtained from the NIR spectra can be used as a quantitative parameter to detect and quantify damage.

Composites' reliability in the industry can significantly improve through further development of this technique. Future work would include deconvolution of the absorbance spectral to separate the absorbed moisture into their free/bound states. More study is needed to improve the technology and apply it to large-scale products to reduce the time it takes to gather spectral data. The NIR is in its emerging stage for use to gather spectral data in composites based on moisture content. The next step would be to better understand and improve the NIR approach and then create a faster, more integrated system that employs a machine learning model to analyze data and forecast damage in composite based on the moisture content.

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