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Application of NMR in Textiles, A Review

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Abstract NMR techniques are widely used in structure determination of newly synthesized materials in textiles. Mainly ^{1}H and ^{13}C NMRs are extensively used in textiles. Authors reported several findings regarding NMR application in textiles. Structures of blended fibres were established by these techniques. Structures of newly synthesized antibacterial agents used in textiles also established by interpretation of NMR spectra. In some articles authors reported structures of naturally occurring dyes by using spectral techniques. A comprehensive literature survey has been done on this topic and discussed in this article.

Keywords NMR, Application, Textiles, Structure, Blend, Antibacterial agent

1. Introduction

Nuclear magnetic resonance spectroscopy deals with nucleus where a perturbation occurs between a strong and a weak Magnetic Field. This phenomenon creates a signal of electromagnetic property at a particular frequency of a magnetic field. This happens near resonance, when oscillation frequency matches the intrinsic frequency of nuclei. It depends on the strength of the static magnetic field. It depends on the magnetic properties of isotope. NMR is extensively used in determination of structures of molecules. It is performed in solution using NMR solvents. It is also used in molecular physics and crystalline along with non-crystalline substances. It is also used in medical science to take image of organs called magnetic resonance imaging. Most common NMRs are ^{1}H and ^{13}C. In NMR technique a constant magnetic field is applied which polarizes nuclear spins belonging to magnetic property. When perturbation occurs in polarization of magnetic nuclear spins is called radio frequency pulse. It is occurred by oscillating magnetic field which is weak in nature. The NMR signal is detected at any certain radio frequency pulse when a voltage is induced around nuclear spins in a detection coil [1]. The NMR spectroscopy has been used in many cases in textiles [2-4].

2. Discussion

Sixtaa et al. reported an effective NMR belonging to a method called solid state for quantification a blend made of cellulose and polyester used in textiles [2]. Authors explained that, NMR method is applied in solid state method for above mentioned quantification which promoted valorization of a textile waste containing cellulose. In this work authors used carbon NMR which is a significant NMR for structure determination in blend made of cotton and polyester. A sigmoidal curve obtained after calibration using Gaussian functions for integration of the spectra which was achieved by means of standards. A method applied as reference called acid hydrolysis. Authors mentioned that this NMR technique is reliable method to determine both of cellulose and polyester fibres in textile wastes before and after involvement of consumers and it is also applicable in blending of synthetic (man-made) fibres.

In an another report, Li and coworkers reported a method to apply both of ^{1}H and ^{13}C NMR techniques to confirm structures of newly synthesized dyes [3]. Authors reported that, new dye was synthesized by reactions using diazo and coupling components. After confirmation of structures of newly synthesized reactive, disperse and reactive disperse dyes by analyzing NMR spectra authors studied dyeing performances on blended fabric.

Otutu et al. reported structures of six newly synthesized disperse dyes by reacting amino and another compounds called hydroxyl derivatives to substituted nitroaniline and dihydroxypyrimidine by coupling with diazo compounds [4]. The structures of bi-substituted azo dyes were confirmed by both of ^{1}H and ^{13}C NMRs. Then synthesized dye was for dyeing of polyester and nylon 6 fabrics. Eventually dyeing performance was analyzed.

In a report authors reported application of NMR techniques in identification of structures of new antibacterial agents which are applied to check antibacterial activity on cotton fabric [5]. At first authors synthesized a complex by silver and abietic acid. The complex structure was confirmed by NMR spectra. The newly synthesized complex did not possess any environmentally detrimental effect. The antibacterial effect of the complex was examined by using agar diffusion method to cotton fabric. Antibacterial effect was stable after several times washing so that it is suitable to

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use as antibacterial agent in textiles.

Ocal et al. reported newly synthesized heterocyclic imines which are useful in textiles [6]. As per report, natural heterocyclic compounds are used as essential drugs for treatment of several diseases like cancer. Authors explained that they synthesized aldimines and established structures by NMR spectra. Authors also mentioned that they dissolved compounds in methelene chloride (also known as dichloromethane) and applied on cotton fabric after mixing with a printing paste. According to the article, it was found that UV was absorbed on cotton fabric after treatment of a mixture of new compounds and printing paste on it. Experimental result showed that transmittance of the radiations decreased after treatment of the new compound through cotton fabric. After washing several times the protective nature from radiation remains active.

In a report, authors reported interesting phenomenon using NMR in textiles [7]. They reported characterization of void structure by using different methods. They proposed that NMR is applicable to establish void structure created by both of interaction and movement of fluids in textile products. In this work authors measured dimensions of average void by NMR imaging in fabric. Authors also found the possibility of analyzing of void space after calculation of density of fluid with a function of autocorrelation. They applied this technique to nylon fabric which was saturated by water with optical microscopy.

Palme and coworkers reported that textile industries are pressurized to decrease pollution load because of production and uses [8]. Recycling is a way to decrease this load. To facilitate recycling of cotton fabric a property called "age-induced" is important and it needs to understand by concerned people. Authors reported two types of changes, chemical and ultrastructural take place in cellulosic cotton fabric because of laundering and long time use. As per report, changes studied by different techniques, NMR is one of those techniques. Authors clearly mentioned that recycling of cellulosic fibre after regenerated is possible which was understood when molecular mass was largely decreased. Authors experimentally proved that after long time use cellulosic reactivity of cotton fabric was decreased because of long time use and laundering.

In an another paper, Palme et al. reported a method for recycling of polyester and cotton components from a fabric made of those fibres [9]. Authors used NMR technique in this work. Authors explained that recycling using chemical method could be a way to get quality materials from textile wastes. Author focused about separation method of polyester (PET) and cotton from textile materials. Authors emphasized on polycotton because it is generally used in towel manufacturing. They used 5-15% aqueous sodium hydroxide solution at 70-90℃ for polyester hydrolysis. In this reaction polyester was converted to its monomers. Terephthalic acid and ethylene glycol are prepared after degradation of polyester. This degradation was identified by several techniques using NMR. As per the paper, catalytic hydrolysis of polyester required only 40 minute to complete the process with 10% aqueous sodium hydroxide solution at 90℃. Experimental results showed that no other substance was present in separated terephthalic acid and ethylene glycol was recovered from filtrate. 97% cellulosic cotton was recovered from the blend.

Colletti and coworker reported use of ^{13}C NMR for rapid characterization of components in textile materials by both of qualitative and quantitative estimations [10]. Minimal preparation of sample was done for a mixture of two fibres, polyester and cotton.

NMR techniques is applicable to characterize dye from natural products. Tayade et al. reported isolation and characterization of indigo dye from a natural product named, *Couroupita guianensis* [11]. Authors reported that, indigotin with indirubin are present in canon ball tree (*Couroupita guianensis*). Authors optimized extraction of indigo dye effectively from the fruit of above mentioned tree. They applied extract on cotton fabric. The optimum time period for fermentation of fruit was 5 days. After 5 days intense blue colored pigment was found. Retardation factor (R_f) of isolated blue color was 0.25 with chloroform. NMR spectrum of blue colored material isolated from the tree matched with standard indigo dye. 26.46% purity achieved in extracted blue colored substance.

In another paper, Mirjalili et al. reported extraction and characterization methods of natural dye form shell of walnut [12]. They used soxhlet extractor for this work. The coloring materials separated by column chromatography and characterized by using several techniques including NMR. The extracted dye was applied on polyamide fabric and dyeing performance achieved well.

Betel nut is a good source of natural dye. Extracted dye from betel nut (*Areca catechu*) could be used for dyeing of silk and nylon fabric. The compound extracted from this natural product was confirmed by spectral analysis which includes NMR [13].

^{13}C NMR is applicable to elucidate supramolecular structure of cellulose. Zuckerstätter et al. reported an article on application of ^{13}C NMR spectroscopy [14]. Authors also established crystallinity morphology and domain sizes of fibre. This investigation helped to influence several properties of cellulose such as mechanical including physical and chemical as per interest of industries. Parameters of physical model were interpreted by NMR spectrum.

3. Recommendations

From the above discussion it is well understood that enough research papers have not been published on this area of research. More works need to be done with NMR application in textiles. In case of synthesis of new textile dyes both of ^{1}H and ^{13}C NMR should be used to elucidate structure. A gap was found in this area of research works. 2D NMR was not used in most of the cases. 2D NMR can play vital role for more accurate structure determination. Further works using 2D NMR are recommended for development of synthetic methods in textiles. Many new natural dyes can be

isolated from naturally occurring substances and structure of newly synthesized compounds can be identified by different types of NMR techniques such as ^{1}H, ^{13}C, ^{31}P, ^{19}F, ^{17}O etc. Another suggestion is, use ^{17}O NMR should be applied for identification of heterocyclic linkages in natural and synthetic dyes.

4. Conclusions

Nuclear Magnetic Resonance (NMR) spectroscopy is a vital method to determine structure of newly synthesized textile materials. By using ^{1}H and ^{13}C NMR techniques structures are elucidated. NMR techniques are effective to establish structures of different types of blend, antibacterial compounds and naturally occurring dyes.

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