



## FTIR Studies on Composite Blends and Biodegradable Products of Guna Protein/Cellulose Based Biocomposites

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### Authors' contributions

This work was carried out in collaboration between all authors. Author AAH designed the study and wrote the protocol. Authors AAH, BAA, SAO and JBT performed the statistical analysis, managed the literature search and wrote the first draft of the manuscript with assistance from authors AAH, BAA and JBT. All authors read and approved the final manuscript.

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### ABSTRACT

Composite blends were formulated from guna protein/cellulose based biomaterials. The blends ratios prepared using solution method to produce blends ratios of (90/10 to 0/100). These were subjected to soil burial tests from 20 days through 80 days. FTIR analysis was carried on the composites and degraded blends. The results obtained from FTIR analysis confirmed the degradability of the polymer (guna protein/cellulose) by chemical changes in terms of appearance and shortening of peaks. Remarkably, peaks were newly indicated at 703 cm<sup>-1</sup> and 861 cm<sup>-1</sup> respectively. These absorption bands were ascribed to C-H aromatic bending and C-H bending

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respectively. Consistency in peaks at a frequency of  $2966\text{ cm}^{-1}$  ascribed to C-O Group was recorded for all the blend ratios. Aldehyde group stretching at a frequency of  $1715\text{ cm}^{-1}$  was also observed for degraded blends.

*Keywords: Biodegradability; FTIR; guna protein; cellulose; composites.*

## 1. INTRODUCTION

The problems of non-biodegradable plastics waste remains a challenge due to its negative environmental impact. In this sense, biodegradable polymers from natural sources have been receiving much attention lately due to their biodegradability in soil, biocompatibility, environmentally friendly characteristics and above all non-toxicity [1].

Biodegradable composites made entirely from natural materials such as corn, wheat, protein etc, are recyclable and no additional energy is required to break down the materials. However, the commonly used synthetic polymers like polyethylene, polypropylene etc are normally non-biodegradable under environmentally triggered condition and persist for a long creating cycles of waste and waste disposal problems [2]. Therefore, uses of green polymeric materials especially renewable polymers and polymer materials are most encouraging from the ecological, economical, environmental protection and as well as resource recycling point of view [3,4].

Biodegradable polymers derived from renewable source have many functional properties; a remarkable property is recorded in packaging as carry bags for delivery of food stuff, clothes, gifts and other perishable commodities. The uses of plastic shopping bags are now a day to day practice in many countries of the world [3,4].

Consequently, growing concerns the world over as regards to the persistence of synthetic polymers have generated increased public and scientific interests regarding the use and development of biodegradable polymers as ecologically useful alternative to synthetic polymers which must still retain the desired chemical and physical properties of conventional plastics, thus providing a solution for the existing severe problems of plastic waste. In this regards, cellulose as an abundant biopolymer in the productions of polymeric materials such as load bearing components, fibres and other applications that require stiffness [2]. Mixtures

can be used as new biomaterials for producing new process able polymeric materials that hopefully possess biocompatibility properties. Polymer blending is designed to produce materials with optimized chemical, structural, morphological, biological as well as mechanical properties [6,4].

For the reasons mentioned above, there is considerable interest to develop environmentally-friendly, fully biodegradable "green" composites using yearly renewable (sustainable) plant-based fibres and resins [7-14].

Results of these investigations revealed that green composites obtained from Guna protein/fabrics and soy protein/yarns are of great improvements in terms of mechanical properties as well as the plausibility of using the composites (soy protein/yarn) for secondary structural applications such as door panels and packaging [15]. The use of other protein source like soybeans for example with 40% protein content with even at a lower functionality of approximately 2.5 [16] showed results that are indicative of increased mechanical properties in terms of modulus suitable for packaging. Guna protein as a biopolymer has 60% [17,18] protein content over soy beans coupled with reported higher functionality suggests better properties in the finished composites. Greater functionality implies possibility of better mixing/blending of composites thereby reducing the problems of phase separation, leading to greater improvement in terms of performance and improved properties.

FTIR is a dependable tool in the determinations of elucidation of molecular structures and applications. Some researchers [19,20] have reported the use of FTIR in study of natural polymers and protein material.

Based upon these considerations, FTIR was used to access the molecular interaction of GP/CF composites in their various blends and degradability in various days.

## 2. MATERIALS AND METHODS

### 2.1 Materials

A plain woven 100% cotton fabric of commercial quality was obtained from the market (Jimeta ultra-modern market). Guna (*Citrillus vulgaris*) is obtained from farm lands in Jimeta.

Assorted chemicals of analytical grade supplied by British drug House (BDH) were used and these included ethanol, acetic acid, potassium dihydrogen phosphate, magnesium sulphate, sodium silicate hydrogen tetraoxosulphate (VI) acid, hydrogen peroxide and sodium hydrogen. Other materials used included distilled, flower pots and pH meter. Also used was SHIMADZU 8400S fourier transformed infra-red spectrophotomer.

### 2.2 Methods

#### 2.2.1 Sample preparation

The method described by [21] as modified by [22] was adopted for the resin preparation. Blends of GPR/CF composite were prepared by firstly blending the modified resin and water in the ratio of 1:10 of Guna protein resin (GPR)/water to obtain various proportions of the blends of water in the proportions 0/100 to 100/0.

#### 2.2.2 Biodegradability studies

Samples of 10 cm in length and 2.5 cm (width) were cut and prepared from all the composite composition and prepared in accordance to the method described in section 2 of (ASTMD790).

The composite blends were buried in composted soil obtained from a farmland filled into pots to their capacities. The soil pots containing the samples were kept in laboratory and moisture maintained by sprinkling moisture at regular intervals. The excess water was drained from a hole at the bottom of the pot.

The degradation of the samples was determined at regular intervals of 20 days by carefully removing the samples from the soil and gently washing them with distilled water to remove the soil from the samples. These were then dried in an air oven at 50°C for 24 h. The samples were then kept in polythene bags until needed for further analysis.

### 2.3 Characterization

Fourier transformed infra-red spectrophotometer (FTIR) SHIMADZU MODEL 8400S with the wave number range of 4500 to 500  $\text{cm}^{-1}$  was used to obtain the spectra of the various ratios of the composites blend and degraded composites at NARICT Zaria driven by a computer. The analysis is carried on both neat and degraded samples and also reference samples for mixture comparison to the qualitative assessment of chemical change. The test analysis using the FTIR is carried on neat samples of both degraded and neat blends without mixing with chemicals to avoid interference. These were mounted on to the instrument and the frequency and % transmittance were automatically plotted and printed out via a computer.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Results

From Table 1 the spectral data indicated bands which could be ascribed to C-H, COOH, Amide1 and 11(N-H) groups which are usually protein assigned bands and are given the nomenclatures used in this text in accordance with intensity of the observed bands, ketone and bands ascribed to Amide 1, carbonyl, ketone and carboxylic groups were registered as reported in the studies of [23,19,24]. Indication of Amide1 band was at a wave number of 1600 $\text{cm}^{-1}$  was registered in control. This band was also replicated in composite ratios 40/60 at a frequency of 1622  $\text{cm}^{-1}$  and ratio 30/70 at wave number of 3550  $\text{cm}^{-1}$  indicative of N-H group stretching which could be attributed to O-H stretch and COO $^{-}$  in ester group which is also reported in the studies of [25]. A band of C=C stretch was observed on the spectra of composite ratios 20/80 and 80/20 arising from the wave number of 1693  $\text{cm}^{-1}$ . There was a band of arising from C=O which was ascribed to carbonyl stretch in the blends which. This is shown in Table 1.

Table 2 represents the FTIR results of degraded composites of various ratios for a period of 20 days. From the spectra, there were indications of carbonyl group stretch at a medium of 1709  $\text{cm}^{-1}$  in all the blend ratios. There was the appearance of two new peaks at the wave numbers of 860  $\text{cm}^{-1}$  and 706  $\text{cm}^{-1}$  in all the blends ascribed to aromatic bending and C-H bending respectively. A peak of high intensity of 2956  $\text{cm}^{-1}$  ascribed to CH<sub>2</sub> stretch was also observed in the sample

blends. There was peak arising from the band of  $1713\text{ cm}^{-1}$  which could be ascribed to carbonyl group or ketone group in all the blends. The intensity of these peaks were however reduced peaks due likely to protein dispersed on the surface of the cellulose material as reported in the studies of [24,26].

**Table 1. Spectral analysis of pure composite blends**

Blend ratio	Spectral bands ( $\text{cm}^{-1}$ )	Tentative assignment of band
100/0	1709	C=O(Stretch)
90/10	1765	C=O(Stretch)
80/20	1693	C=O(Stretch)
70/30	3550	N-H(Amide)
60/40	1621	C=O(Stretch)
50/50	1600	N-H(Amide)
40/60	1623	C=O(Stretch)
30/70	1600	N-H(Amide)
20/80	1693	C=O(Stretch)
10/90	1754	C=O(Stretch)
0/100	1785	C=O(Stretch)

There was a peak at  $1033\text{ cm}^{-1}$  which could be ascribed to C-H group alkane stretch observed in the blend ratio 50/50. This was however observed in the ratio 0/100 double peak at increased intensity arising from a band of  $1117\text{ cm}^{-1}$  which could be ascribed as to C-H group. This shown below in Table 2.

The FTIR spectra of samples subjected to burial period of 40 days is shown in Table 3 below. From the table, there were two new peaks at a reduced intensity of  $695\text{ cm}^{-1}$  and  $861\text{ cm}^{-1}$  which could be ascribed to aromatic C-H stretching and C-H bending respectively. There was a medium band at  $1714\text{ cm}^{-1}$  in samples ratios 70/30, 40/60 which could be ascribed to carbonyl group stretching. Likewise, a high intensity band at a wave number of  $2929\text{ cm}^{-1}$  which could be ascribed to  $\text{CH}_2$  stretch was observed on composite samples of 100/0.

Table 4 above shows the FTIR spectra data of degraded composite samples ratios 100/0 to 0/100 buried for 60 days. From the figures, a peak of  $1712\text{ cm}^{-1}$  for ratio 40/60 and on 0/100 ratio at a wavelength of  $1715\text{ cm}^{-1}$  ascribed to amide N-H group stretch was detected.

A wave number of  $2965\text{ cm}^{-1}$  was observed which is present in other sample ratios investigated and could be ascribed to C=O stretch. Two new wave numbers  $703\text{ cm}^{-1}$  ascribed to C-H aromatic

stretch and  $861\text{ cm}^{-1}$  ascribed to C-H bending were mostly observed in the entire blends.

**Table 2. Spectral analysis of degraded blends buried for 20 days**

Blend ratio	Spectral bands ( $\text{cm}^{-1}$ )	Tentative assignment of band	
100/0	1706	C=O(Ketone Stretch)	
	1028	C-O(Ketone Stretch)	
	846	C-H (Bending)	
	1396	O-H( Bending)	
	2956	$\text{CH}_2$ (stretch)	
90/10	1713	C-H(Stretching)	
	864	C-H(Stretching)	
	1714	C=O(Stretching)	
80/20	1714	C=O(Stretching)	
	795	C-H(Stretching)	
	1117	C-H(alkane stretch)	
70/30	1714	C=O(Stretching)	
	794	C-H(Stretching)	
	854	C-C(Stretching)	
60/40	1714	C=O(Stretching)	
	849	C-C(Stretching)	
	1033	C-O(stretch)	
50/50	1714	C=O(Stretching)	
	849	C-H(Bending)	
	30/70	863	C-H(Bending)
20/80	863	C-H(Bending)	
	10/90	863	C-H(Bending)
	0/100	863	C-H(Bending)

There was a peak arising from a wave number of  $1714\text{ cm}^{-1}$  which could be ascribed to amide N-H group stretch and another sharp peak arising from a wave number of  $2966\text{ cm}^{-1}$  ascribed to C=O stretch was observed in all the samples. A further observation showed two new peaks at  $703\text{ cm}^{-1}$  ascribed to C-H aromatic stretch and the other at  $861\text{ cm}^{-1}$  ascribed to C-H ring bending was observed in the entire samples.

The extracted spectral bands of biodegraded composite samples ratios 100/0 to 0/100 for 80 days are shown in Table 5 below.

From Table 5, it was observed that there was an appearance of a new peak at a wave number of  $1110\text{ cm}^{-1}$  which could be ascribed to C-O stretch on the blend ratio 40/60. Also a band at  $2961\text{ cm}^{-1}$  ascribed to O-H stretching was observed.

There was a peak at  $1715\text{ cm}^{-1}$  ascribed to C=O ketone, also observed were consistent appearance of two new peaks of weak intensity at  $707\text{ cm}^{-1}$  and  $854\text{ cm}^{-1}$  ascribed to C-H aromatic stretch and C-H alkane bending respectively in

composite ratio 30/70. This could be attributed to the onset of degradation which could lead to rupturing of the C-H bonds in the blend 30/70 due to presence higher amount of Guna protein (GP). This may have resulted in the creation of a large surface area for micro-organisms to act on the composite blends as reported in the studies of [25].

**Table 3. Spectral bands of degraded blends buried for 40 days**

Blend ratio	Spectral bands (cm <sup>-1</sup> )	Tentative assignment of band
100/0	1034	C=O (Stretch)
	1435	C-H (Bending)
	2929	CH <sub>2</sub> (Stretch)
90/10	1715	C=O (Ketone Stretch)
	1118	C-H (Bending)
	704	C-H(Bending)
	863	C-H (Bending)
80/20	1703	C=O (Ketone Stretch)
70/30	863	C-H (Alkane Stretch)
	704	C-H (Stretching)
	1715	C-H (Bending)
60/40	1118	C=O (Ketone Stretch)
	1704	C-H (Alkane Bending)
	50/50	1707
40/60	1034	C=O (Ketone Stretch)
	864	C=O (Ketone Stretch)
	1713	C-H (Alkane Stretch)
	1115	C-H (Alkane Bending)
	1032	C=O (Ketone Stretch)
30/70	865	C-H (Alkane Stretch)
	1714	C-H (Bending)
	1114	C=O (Ketone Stretch)
	1593	C-H (Alkane Stretch)
	1029	C-O (Stretch)
20/80	695	C-H (Stretching)
	861	C-H (Bending)
	803	C-H (Bending)
	704	C-H (Stretching)
	1024	C-H (Alkane Stretch)
10/90	1118	C-O (Stretch)
	1715	C=O (Ketone Stretch)
	695	C-H (Stretching)
	862	C-H (Bending)
	1716	C=O (Ketone Stretch)
0/100	1580	N-H (Amide Bend)
	1110	C-H (Alkane Stretch)
	1709	C=O (Ketone Stretch)

There was also the presence of peaks at the wave-numbers 2111 cm<sup>-1</sup> and 2264 cm<sup>-1</sup> ascribed to C-H alkane stretch in the entire blends. The peak at wave number of 2264 cm<sup>-1</sup> could be attributable to asymmetric stretching vibration of

CH<sub>2</sub> closely followed by second peak attributable to symmetric vibrations reported in the studies by [26,25]. The bending and wagging vibrations of CH<sub>2</sub> has raised the wave numbers or bands in all other blends. This is shown in Table 5.

**Table 4. Spectral analysis of degraded blends buried for 60 days**

Blend ratio	Spectral bands (cm <sup>-1</sup> )	Tentative assignment of band	
100/0	1710	C=O (Amide Stretching)	
	854	C-H (Bending)	
	603	C-H (Bending)	
90/10	1035	C=O(Ketone Stretch)	
	1435	C-O (Stretching)	
80/20	785	C=C (Aromatic Ring Stretch)	
	854	C-H (Stretching)	
	854	C-H(Bending)	
	1114	C-H (Alkane Stretch)	
	1711	N-H (Amide Stretching)	
70/30	704	C-H (Stretching)	
	803	C-H (Bending)	
	1118	C-H (Stretching)	
60/40	854	C-H (Alkane Stretch)	
	603	C=O (Ketone Stretch)	
	1117	C-H (Stretching)	
	1706	C=O (Ketone Stretch)	
50/50	1037	C=O (Ketone Stretch)	
	40/60	1716	C=O (Ketone Stretch)
	703	C=O (Ketone Stretch)	
30/70	861	C-H (Alkane Bending)	
	1113	C-H (Alkane Stretch)	
	702	C-H (Stretching)	
	860	C-H (Alkane Bending)	
	1715	C=O(Ketone Stretch)	
20/80	1025	C-O (stretch)	
	1035	C=O (Ketone Stretch)	
	10/90	1712	C=O (Ketone Stretch)
10/90	1115	C-H (Alkane Stretch)	
	802	C-H (Bending)	
	678	C-H (Plane Stretch)	
	1710	N-H (Amide Stretching)	
	855	C-H (Bending)	

In all the spectral data, the degradation of GP/CF composites is indicated by the band intensity corresponding to C-H bending deformational vibrations in CH<sub>3</sub> groups ((1714, 802 cm<sup>-1</sup>). The intensities of C=O stretch show decrease after biodegrading (1785, 1714 cm<sup>-1</sup>). The changes in the C=O band are associated with changes in the number of carboxylic end groups in the polymer chain during hydrolytic degradation as

well as cycles of microbial attack [27]. There was also a shift in intensity to a higher wavelength of N-H group ( $1600, 1715 \text{ cm}^{-1}$ ) structure which is also reported by [23]. The changes in the number of carboxylic groups that may have resulted from the changes in C-H stretching as the days of degradation slightly increased. This indicate that the cellulose may have ruptured, therefore paving the way for organisms to attack the chain easily [19,21]. Two peaks which were originally present ( $861, 703 \text{ cm}^{-1}$ ) characteristics of plane stretch and bending respectively slightly increased as the days of degradation.

**Table 5. Spectral analysis of degraded blends buried for 80 days**

Blend ratio	Spectral bands ( $\text{cm}^{-1}$ )	Tentative assignment of band
100/0	1716	C=O (Ketone stretch)
	859	C-H (Alkane stretch)
	704	C-H (Bending)
90/10	1714	C=O (Ketone Stretch)
80/20	1714	C=O (Ketone Stretch)
70/30	1715	C=O (Ketone Stretch)
	704	C-H (Stretching)
	854	C-H (Stretching)
60/40	1713	C=O (Ketone Stretch)
50/50	1036	C-O (Stretch)
40/60	1713	C=O (Ketone Stretch)
30/70	1712	C=O (Ketone Stretch)
20/80	850	C-H (Bending)
	673	C-H (Stretch)
	1711	C=O(Stretch)
	1116	C-H (Alkane Stretch)
	847	C-H (Bending)
	680	C-H (Stretching)
10/90	1727	N-H (Amide Stretching)
0/100	1713	N-H (Amide Stretching)
	1117	C-H(Alkane Stretch)
	1025	C-H(Alkane Stretch)

In contrast to un-degraded blends, the degraded blends, showed bands at  $1026$  to  $1038 \text{ cm}^{-1}$  (C-O stretching/side-chain vibrations) as clarified by the following; in 20 days of degradation (50/50 blend at  $1033 \text{ cm}^{-1}$ ) in 40 days of degradation (50/50 at  $1034 \text{ cm}^{-1}$ , 30/70 at  $1029 \text{ cm}^{-1}$  and 20/80 at  $1024 \text{ cm}^{-1}$  respectively), in 60 days of degradation (50/50 at  $1037 \text{ cm}^{-1}$ , 30/70 at  $1025 \text{ cm}^{-1}$ ) and lastly at 80 days of degradation in the blends (50/50 at  $1035 \text{ cm}^{-1}$ , 0/100 at  $1025 \text{ cm}^{-1}$ ). The shifts in most peaks towards higher wave-numbers may signify the shortening of polymer bonds finally leading to rupturing of polymer

composite bonds. Thus, the FTIR studies of the neat and buried samples suggest strongly that some chemical changes have occurred in some cases indicative of microbial attack and therefore samples prepared are amenable to biodegradation although more work is required.

#### 4. CONCLUSION

Collecting the above results of the investigations carried out on the GP/CF composite, the following conclusions could be made:

- (i). The FTIR measurement for neat blends have been observed to show peaks at the wave numbers of ( $1785-1715 \text{ cm}^{-1}$ ), which signifies the formation of esters between the O-H groups of cellulose and COOH of the Guna protein, showing not only physical but also chemical changes. These values however, increased in blends as the compositions and days of burial tests were varied.
- (ii). The increase of peaks of ( $1714-1600 \text{ cm}^{-1}$ ) in pure blends and subsequently in degraded blends also signifies degradation.
- (iii). Chemically modified biomaterial Cellulose, used in this research work has enhanced biodegradation of bio-composite produced that is CF/GP composite. The results in this study therefore demonstrated the efficiency of the modification of cellulose as an efficient means in enhancing biodegradability.
- (iv). The biopolymers used in this research work can be harnessed in sustainable manner for composite production. It has been reported that the use of biopolymers in composites production is of great advantage over conventional or fossil based sources. This is due to the fact that the plant (*Citrillus vulgaris*) is an all year crop grown in most parts of the north and west of Nigeria and even neighboring countries and states.

The development of bio-composite in a sustainable manner is still a grey area in the research community. If the technology is to be put to use, then the main focus of bio-composite is the biodegrading of both the matrix and the reinforcement phases. This together with compatibility of the matrix phase and reinforcement observed in this work leads to the success of producing a complete biodegradable composite as observed in the final product.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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