

Effects of Microwave Irradiative Processing On the Mechanical Properties of Gum Arabic-Based Films

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Abstract: Each of 5,10,20,30 and 50% aqueous solution of Gum Arabic(GA) was exposed in turn to microwave irradiation power at 100,200 and 300W for 1, 2 and 3 minutes respectively. Dried to constant weight film of each was obtained by casting method and then subjected to characterization by determination of pH, specific gravity(SG), tensile strength(TS), Young's modulus(YM), elongation at break(EB) and energy at break(EAB). The results showed that increasing microwave irradiation power for longer time was accompanied by increase in processing temperature as concentration of the biopolymer increased. These resulted in decreased solution pH, increased SG, and generally enhanced GA film mechanical properties up to the extent of increasing the TS five and half times, YM twenty six times, and as expected the EB reduced even to twenty times, using 30%, two separate 50% GA solutions exposed to microwave radiation of 300W for 3 minutes, 200W for 1 minute and 200W for 2minutes respectively along with greater EAB(toughness) relative to the corresponding properties of the control(unexposed) samples. Further aspects of this study showed that the presence of TiO₂ formulated with in irradiated 50% GA solution gave additional improvements in the mechanical properties over those of the neat and non-pigmented samples.

I. Introduction

Microwave irradiation (MWI) is a form of energy in the electromagnetic spectrum with relatively low frequency of 300 to 300,000 megahertz (MHz).. Therefore it will only cause bonds to rotate, not break unlike radiations such as UV or X-rays.(Hayes, 2002).Therefore, exposure of materials or reactants or materialsto MWI will not cause direct chemical reactions to occur but can lead to rotation and orientation of molecules for effective packing/alignment of polar molecules resulting into more crystalline material kinetically and thermodynamically. Thus MWI has been employed in organic /inorganic synthesis, polymer synthesis and food processing in polar media. In general, if a high temperature is required, a very polar solvent can be used to reach this high temperature very rapidly. Lower absorbing solvents, however, can still be used to reach high temperatures because, in most cases, the starting materials, catalyst, or other reaction components will absorb microwave energy (Hayes, 2002; Loupy, 2002; Oespchuck., Liao (2002) .

Many researchers recognized that GA, a highly heterogeneous material, can be separated into three major fractions by hydrophobic affinity chromatography(Randall et al., 1989).The major fraction is a highly branched polysaccharide consisting of β -(1 3) galactose backbone with linked branches of arabinose and rhamnose which terminate in glucuronic acid found in nature as magnesium, potassium and calcium salts (Renard et al., 2006, Sanchez et al., 2008). A smaller fraction is a higher molecular weight arabinogalactan-protein complex (AGP-GA glycoprotein) in which arabinogalactan chains are covalently linked to a protein chain through serine and hydroxyproline groups. The attached arabinogalactan in the complex contains glucuronic acid (Goodrum et al., 2000). Lastly, the smallest fraction having the highest protein content is a glycoprotein which differs in its amino acids composition (Anderson and Stoddart, 1996; Verbeken et al., 2003),AlAssaf, et al., 2005 a,b; Flindt et al., 2005; Hassan et al., 2005;Karamalla et al., 1998; Siddig et al., 2005). Both Acacia Senegal andAcacial Seyal are the common GA. Acacia Seyal gum contains a lower proportion of nitrogen, and specific rotations are also completely different. The determination of the latter parameters may clearly spot the difference between the two species (Osman et al., 1993),Eqbal, et al., 2013).Studies on the physico-chemical properties have revealed that GA has high water solubility up to a concentration of 50% and relatively low viscosity with an acceptable pH range of 4.5 ± 0.21 at 25⁰C(Karamallah et al.,1998.,Younes,2009).

Gum Arabic has found extensive applications in food and non-food areas. These include its use as stabilizer, thickener, an emulsifier, encapsulating medium and as in confectionery, bakery, diary and beverage in the food industry and to a lesser extent in textiles, ceramics, lithography, cosmetics and pharmaceutical industry(Mariana et al.,2012).

The major interest in this study is based on the need to enhance the mechanical properties of GA which has not been reportedly studied within the literature available to us. It is envisaged that this will expand the technology base of this readily available, eco-friendly/non-toxic and relatively cheap biopolymer.

II. Experimental

Materials

Acacia Senegal (gum Arabic) grade 1 was obtained from Kasunwa-Kurmi market, Kano-State, Nigeria. Distilled water, TR92 (Titanium dioxide pigment), Microwave machine (MASS-II Sineo microwave Chemistry Technology Co-Limited), Pocket-sized pH Meter HANNA Instruments, Specific gravity bottle (25cm³), mechanical stirrer, vacuum pump/desiccator, Buchner flask/funnel, and an improvised glass mould.

Gum Arabic (GA) Processing

Purification and Preparation of Stock solutions of Gum Arabic.

The raw GA were broken into smaller pieces using clean hammer and all the contaminants such as leaf dirt, barks, and stones, were removed manually (hand-picked) and a relatively pure smaller pieces of GA were obtained for faster dissolution in water. The homogeneous low viscous GA liquid obtained was then filtered to remove all the suspended fine particles using vacuum filtration. Neat GA film was obtained by casting on clean glass sheet. Calculated mass of GA equivalent to 5, 10, 20, 30 and 50 % solution, each in 500cm³ volumetric flask was prepared as stock solution.

Exposure of GA solution to Microwave Irradiation

Having prepared the five different concentrations of GA solution, each of them was exposed to microwave irradiation of three powers (W) and exposure time (s) as follows:

60cm³ was measured from the 5% concentration into a 100cm³ flask and exposed to microwave irradiation at 100W for 1, 2 and 3 min(s) separately and then poured into a clean well labeled 100cm³ sample bottle. This was repeated for irradiations at 200W and 300W separately for the same minute(s) as with the 100W. In each case, temperature of the machine was set to the boiling point of the solvent (distilled water) (100°C) and the equilibrium temperature was read off at the end of each exposure time. The above procedure was repeated in turn for the 10%, 20%, 30% and 50% samples.

pH Measurements

The pH of each sample was measured using pH meter (HANNA Instrument, pocket size). This was done by first standardizing the pH meter using prepared standard buffer (KHP/KH₂PO₄-Na₂HPO₄·12H₂O), for pH 4.010 ± 0.010 and pH 7.000 ± 0.010 respectively, at 25°C. These standard solutions were used for standardizing the pH meter at 25°C using a pH temperature correlation table provided by Radiometer Analytical A/S Krogshøjvej 49, DK 2880 Bagsvaerd. The measurements were taken at equilibrium in triplicates and the mean calculated.

Specific gravity determination

The specific gravity of each samples was determined using the 25cm³ SG bottle in triplicates and average calculated.

Preparation of GA Films for, and determination of, Mechanical Properties

20cm³ was measured from each of the GA samples and poured separately into well-labeled glass molds (8x6x0.3cm) on a plumed horizontal concrete slab at 180⁰.

The samples were touch-dried at room temperature. At this point the film obtained possesses some elastic property which makes it easily removed from the glass substrate. The films were removed and a rectangular shape (6cm x 2cm) of approximately equal thickness (using micrometer screw gauge, BS) films were cut out for the mechanical tests. The mechanical properties were determined using a computerized Instron tensile testing machine Model 3304 at the Centre for Energy Research and Development [CERD], Obafemi Awolowo University, Ile-Ife, Nigeria. The cross-head speed was kept at 5mm s⁻¹ at maximum load of 120kg during all the tensile tests.

The Tensile Strength (TS), Young's Modulus (YM), Elongation at Break (EB %) and Energy at Break (EAB (J)) were determined as detailed elsewhere (Turoti et al., 1999)

III. Results And Discussion

Physico-Chemical Effect

Table 1 shows that before exposure to microwave irradiation the pH essentially decreases (more acidic) as concentration of GA increases in line with earlier work (Karamallah et al., 1998). The

Table 1: Effect of Microwave Irradiation and Time on Ph of Gum Arabic Solutions

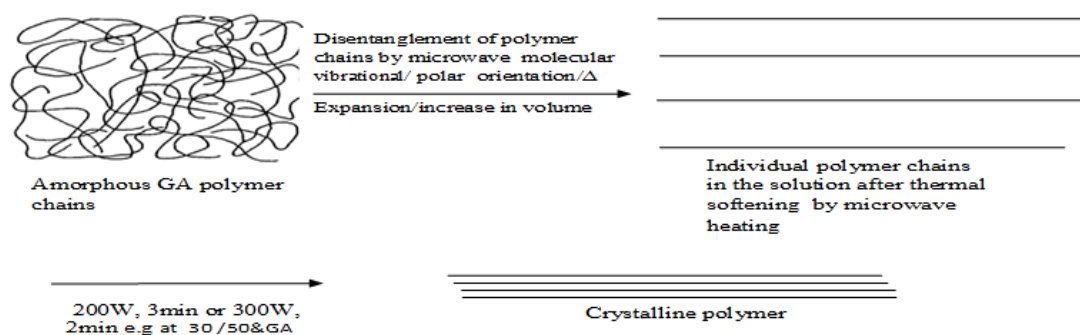
			Concentrations				
			5%	10%	20%	30%	50%
Non-Microwave			4.6	4.4	4.1	4.0	4.0
Microwave	100W	1min	4.8	4.3	4.1	4.1	3.9
		2min	4.7	4.2	3.9	4.1	3.9
		3min	4.5	4.3	3.9	4.1	4.0
	200W	1min	4.8	4.3	3.9	4.1	3.9
		2min	4.5	4.4	4.0	3.9	4.0
		3min	4.5	4.4	4.0	3.9	3.9
	300W	1min	4.9	4.3	3.9	4.0	3.9
		2min	4.6	4.4	4.0	3.9	3.9
		3min	4.6	4.4	4.2	3.9	4.0

Slightly acidic nature of aqueous GA solution is due to the presence of few free carboxyl groups of its constituent acidic group of the D-glucuronic acid and its 4-O-methyl derivatives (Younes, 2009). Similar trend was virtually maintained after irradiation. The decrease in pH suggests that MWI effects evaporation of the polar water solvent, rotate and aligns the coiled chains of the amorphous GA by disentangling them (Hayes, 2002., Loupy, 2002).. These chains contain polar groups including the –COOH making them more available for acidic responses. Furthermore, it can be seen in Table 1 that the solutions become more acidic with greater MWI power and exposure time. Table 2 shows the average specific gravity of each aqueous GA solution for the unexposed and those exposed to microwave irradiations. It was observed that the specific gravity is concentration dependent. That is, as the concentration increases, specific gravity also increases and that the GA solution will tend to become more viscous at higher concentrations. These results show that as MWI wattage and exposure time increase the SG generally increases with increasing concentration.. A most probable explanation for these could be based on the two operational modes of MWI. Thermal evaporation of the aqueous medium is more pronounced the greater the amount of the polar solvent component which is more responsive to microwave.

Table 2: Effect of Microwave Irradiation on Specific Gravity of Gum Arabic Solutions.

Concentration	Specific Gravity
5%, non-microwave	1.01857
100W, 1min	1.01987
100W, 2mins	1.01915
100W, 3mins	1.01930
200W, 1min	1.01865
200W, 2mins	1.01926
200W, 3mins	1.01877
300W, 1min	1.01835
300W, 2mins	1.01869
300W, 3mins	1.01934
10%, non-microwave	1.03807
100W, 1min	1.03745
100W, 2mins	1.03726
100W, 3mins	1.03711
200W, 1min	1.03707
200W, 2mins	1.03719
200W, 3mins	1.03715
300W, 1min	1.03776
300W, 2mins	1.03765
300W, 3mins	1.03795
20%, non-microwave	1.07334
100W, 1min	1.07139
100W, 2mins	1.07181
100W, 3mins	1.07216
200W, 1min	1.07147
200W, 2mins	1.07185
200W, 3mins	1.07204
300W, 1min	1.07093
300W, 2mins	1.07327
300W, 3mins	1.07372
30%, non-microwave	1.10889
100W, 1min	1.10082
100W, 2mins	1.10392
100W, 3mins	1.10862
200W, 1min	1.10625
200W, 2mins	1.10568

200W, 3mins	1.11504
300W, 1min	1.10613
300W, 2mins	1.11003
300W, 3mins	
50%, non-microwave	1.18250
100W, 1min	1.17921
100W, 2mins	1.17951
100W, 3mins	1.17997
200W, 1min	1.18387
200W, 2mins	1.18292
200W, 3mins	
300W, 1min	1.18441
300W, 2mins	1.18502
300W, 3mins	



Scheme 1: Microwave Action On Gum Arabic Chains In Solution.

concentration. However, the greater the concentration of this biopolymer, that is the less the amount of the polar solvent the greater the overall rotational oscillatory action of the microwave and the more the alignment of the GA chains towards each other in compliance with the dipoles of the microwave.

This will result in more compactness of the chains, that is, more crystallinity with the concomitant increase in SG. Thus for 5% concentrations an increase in specific gravity value was noticed which is as a result of escape of water molecules in vapor form from the solutions exposed to irradiations. This is because water the major component of these 5%GA solutions has greater dipole moment than the GA molecules and will be more properly aligned with the electric field of microwave dipole absorbing greater energy which in turn leads to a rise in temperature responsible for loss of water as vapor (Hayes, 2002).

The slight decrease in SG with MWI obtained for each of the 10-30% GA samples could probably be attributed to the state in which the polymer chains indicating that they have not crystallized enough. They might probably be separating through the disentanglement of the chains during which the available energy is being directed mainly for polymer bulk expansion resulting in increasing volume and hence lower SG values. The extent to which this expansion reaches depends on the temperature and concentration of the GA solution.

Thermal behavior of GA Solution

Preliminary investigation shows that temperature of all the experimental GA solutions tends to increase with time of exposure up to the boiling point of water. for each microwave power used in this study. Figures 1 and 2 show the effects of exposure time on the temperatures of the least, 5% GA solution typical of the

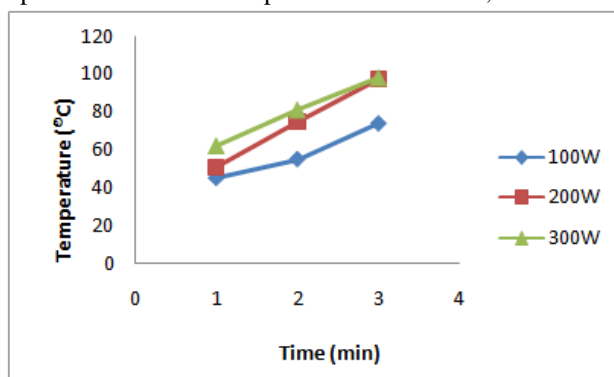


Fig.1: Effect of Exposure Time on Temperature during Microwave Heating At 100W, 200W and 300W Using 5%GA Solution

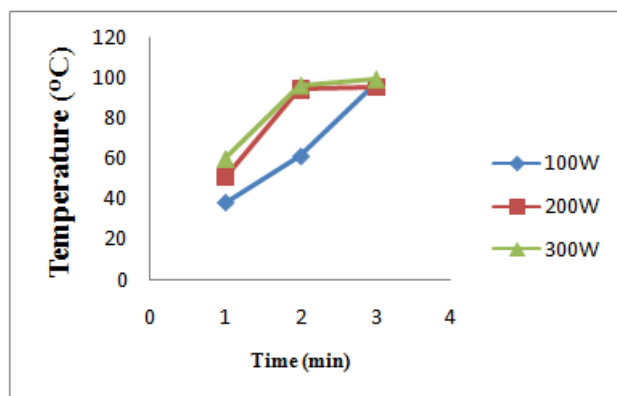


Fig.2: Effect of Exposure Time on Temperature during Microwave Heating At 100W, 200W and 300W Using 50%GA Solution

lower concentrations(5-20%) and the highest 50% GA typical of the higher levels(30-50%) respectively. The results show that temperature of solutions of this biopolymer increases with time and MIP in line with earlier studies (Chen-Chih, 2012; Zhu et al., 2003). Increase in both the concentration of GA and microwave power increase the temperature.

As from Figure 3 the control samples are the GA samples of the same GA concentration that were exposed to microwave irradiation but all stirred mechanically at the same speed for 100minutes each as in earlier work (Turoti et al, 2010). The figures show that rapid rise in temperature essentially depends on the exposure time as it can be seen that in all concentrations, higher temperature is experienced with the samples exposed to microwave irradiation at 100W for longer time (3min). This is in line with the earlier similar works of Kormin et al., 2013; Zhu et al. 2003.

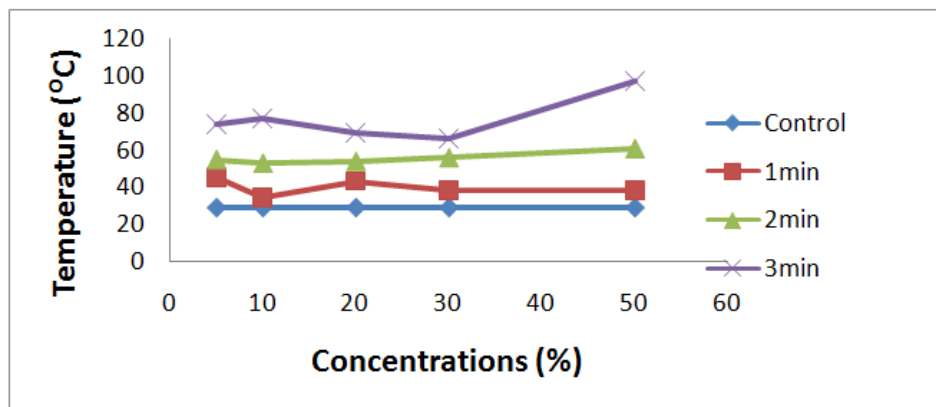


Fig. 3: Effect of concentration on temperature during microwave heating at 100W for varying exposure time.

It can also be seen that exposure of higher concentrations (30% and 50%) to microwave irradiation for short time (1min) is associated with low temperature rise. This is because viscosities at 100W for varying exposure time of these higher concentrations are relatively high and it will take longer time, under constant microwave irradiation, to absorb enough energy that will result in greater temperature rise as viscosity affects the rate of heat transfer. This is in good agreement with the statement of “viscosity affects how a product will flow and in general, a higher viscosity reduces the rate of heat transfer and hence produces more uneven heat distribution” George, (1993).

The 1 minute curve almost assumes a constant temperature as seen in the case of curve for control. This suggests the first transition state temperature of the GA solutions where the disentanglement as well as the stretching out of the coiled or twisted part of the polymer chains occur during exposure to irradiation.

This might cause breaking of some networking (inter-chain) bonds such as H-bonds, weak van der waal forces which results in free flow (movement) of individual polymer chains (Aklonis, 1981;Manjula et al., 2009). At this temperature any additional input energy will be used in breaking these bonds which makes the temperature to be essentially constant.

In the case of the one exposed for 2minutes, an almost linear increase in temperature with concentration was observed. This is because as the concentration increases its thermal retention ability (thermal

capacity, which is the ability to retain the absorbed heat energy) also increases (Buffler, 1991). Therefore high temperature rise was observed in GA solutions of high concentration, with 50% concentration rated as the concentration having greatest thermal capacity among others.

For those exposed for 3minutes, a decreasing trend in temperature was noticed from 10% to 30% concentration and high temperature again for 50% concentration. From this it could be assumed that the 20% and 30% are again reaching the first transition temperature at about 60°C.

Figures 4 and 5 show the effect of concentrations of GA solutions exposed to microwave irradiation on temperature at 200 and 300W for varying exposure time respectively.

From this figure it can be established that temperature rise also depends very much on microwave irradiation power, as it can be seen in all cases that higher temperature was attained compared to their corresponding counterparts in fig. 1.

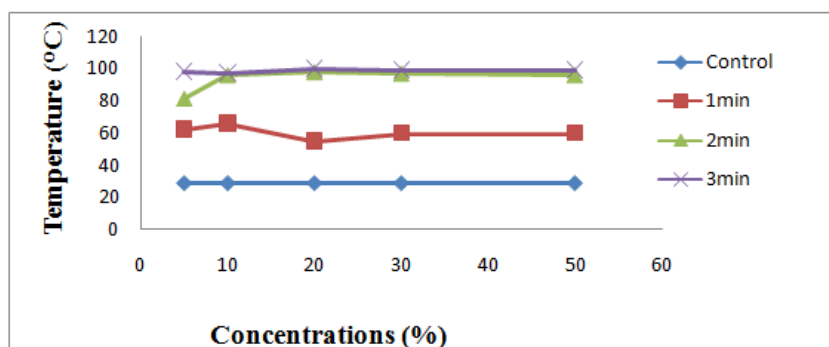


Fig. 4: Effect of concentration on temperature during microwave irradiation at 200W for varying exposure times.

This agrees well with earlier similar works of Alvin et al., (2009) and Chen-Chih,(2012). It can therefore be suggested that the first transition temperature for 5% and 10% concentrations is around 45°C, while those of higher concentrations (20%, 30% and 50%) is around 60°C and the second transition temperature for 5% and 10% concentrations is somewhat around 80°C while those of higher concentrations is around 97°C as the microwave heating continues.

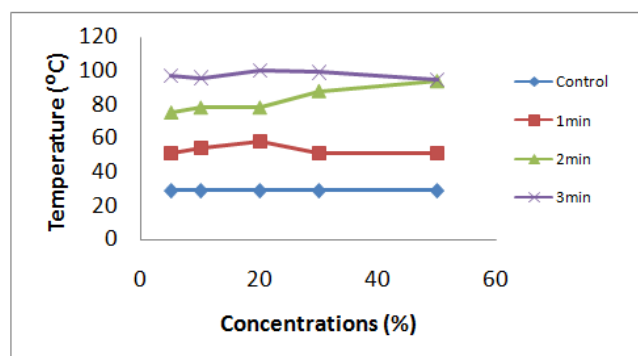


Fig. 5: Effect of Concentration on Temperature during Microwave Heating At 300W for Varying Exposure Times.

It is clear from the previous sections that the concentration of GA solution, time of exposure to microwave irradiation, and power of microwave as well as processing temperature are the most likely parameters that would significantly affect the physical and possibly the mechanical properties of GA films.

Mechanical Properties of Gum Arabic Film

The mechanical properties of 5% and 10% concentrations were not determined. This is because of the limitation encountered in forming the films (very thin and partly broken films were consistently obtained) due to low viscosities. Therefore mechanical test results and discussions were conducted on the films of the remaining three concentrations (20%, 30% and 50%).

Figure 6 shows the effect of microwave power on the tensile strength (TS) of GA films whose solutions are processed by exposure to microwave irradiation at 100W for 1 minute. The films obtained from 50%GA solutions of both the unexposed (control) 50% exposed at 300W have the greatest TS of 4.513 and 17.904

$\times 10^5 \text{Nm}^{-2}$ respectively. The exposed value is almost four times that of the control. Similar plots in Figure 7 for 2minutes exposure also demonstrates the enhanced TS property by the film obtained from 50% GA solution irradiated with 100W having value of $21.898 \times 10^5 \text{Nm}^{-2}$ (about five times that of the control) .

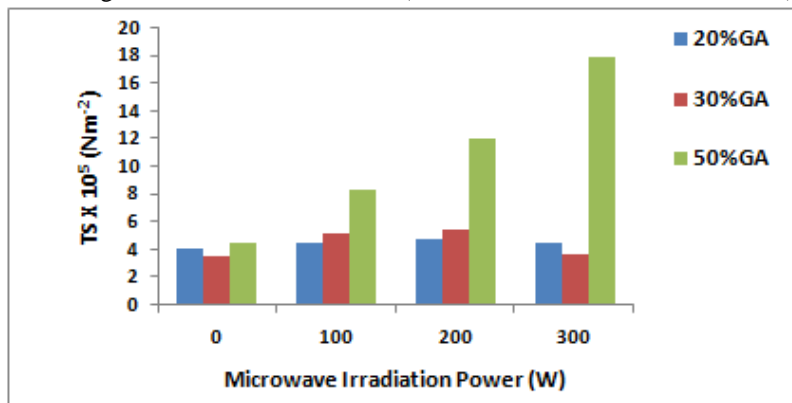


Fig.6: Effect of Microwave Irradiation Power on Tensile Strength of Film from Gum Arabic Exposed For One Minute

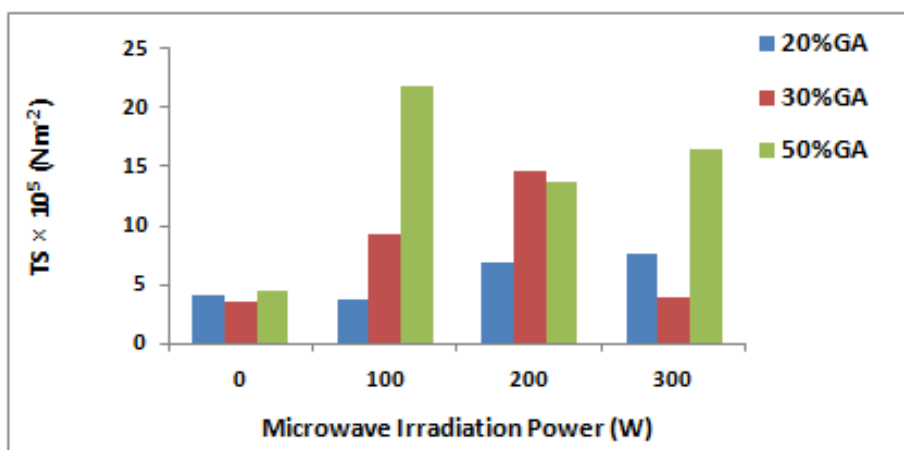


Fig 7: Effect Of Microwave Irradiation Power On Tensile Strength Of Film From Gum Arabic Exposed For Two Minutes.

Figure 8 shows that 30% GA solution irradiated with 300W for 3minutes gives film that has the most enhanced TS value of $25.066 \times 10^5 \text{Nm}^{-2}$ five and half times that of the unexposed. Figures 9-11 show corresponding plots for the Young’s modulus (YM) in

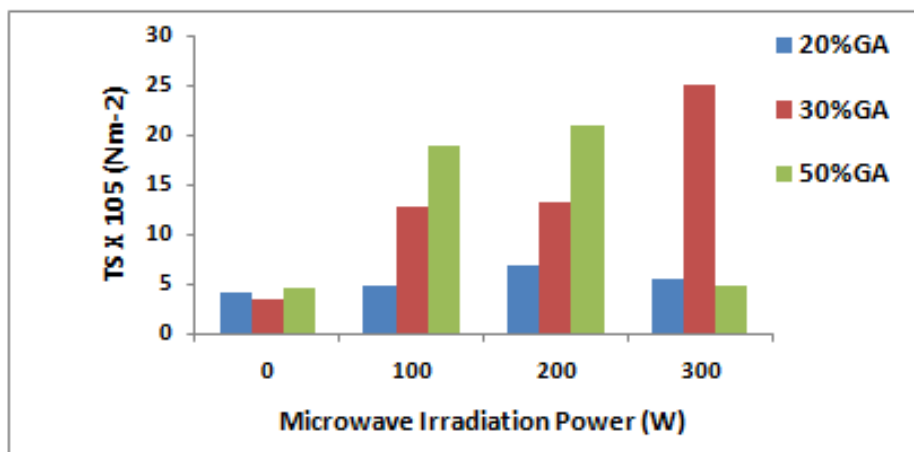


Fig.8: Effect of Microwave Irradiation Power on Tensile Strength of Gum Arabic for Three Minutes.

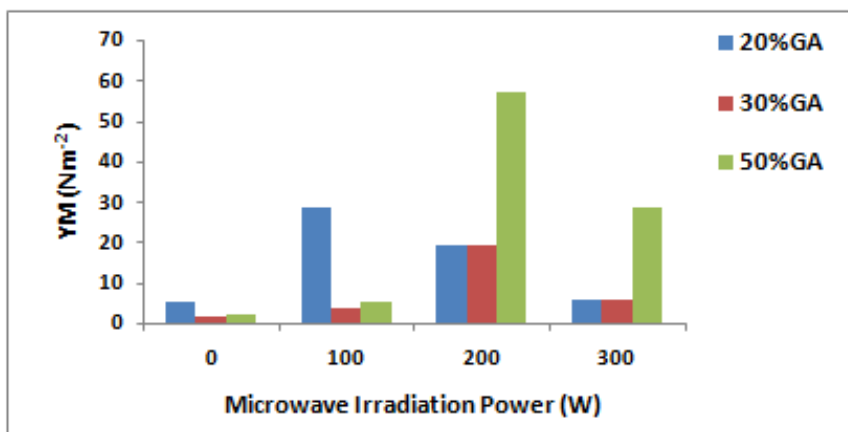


Fig.9: Effect Of Microwave Irradiation Power on Young's Modulus of Gum Arabic for One Minute.

same order as for the TS presentation, for 1, 2 and 3 minute(s) respectively. It is clear that the order of decreasing values of the most enhanced YM values is: 57.29 (50%GA, at 200W), 28.64 (20%GA at 100W) and 28.64 (50%GA at 300W) Nm^{-2} for 1, 2 and 3 minutes respectively. These are about eleven, five and five times that of the greatest YM value of the unexposed of $5.40Nm^{-2}$ obtained from 20%GA solution.

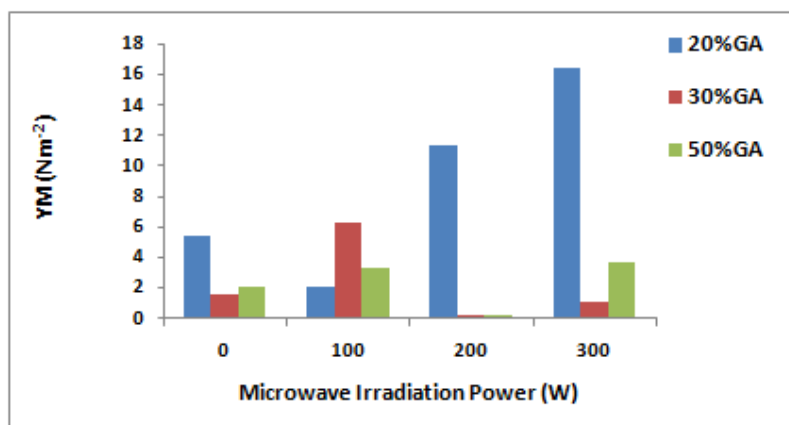


Fig.10: Effect Of Microwave Irradiation Power on Young's Modulus of Gum Arabic Exposed for Two Minutes.

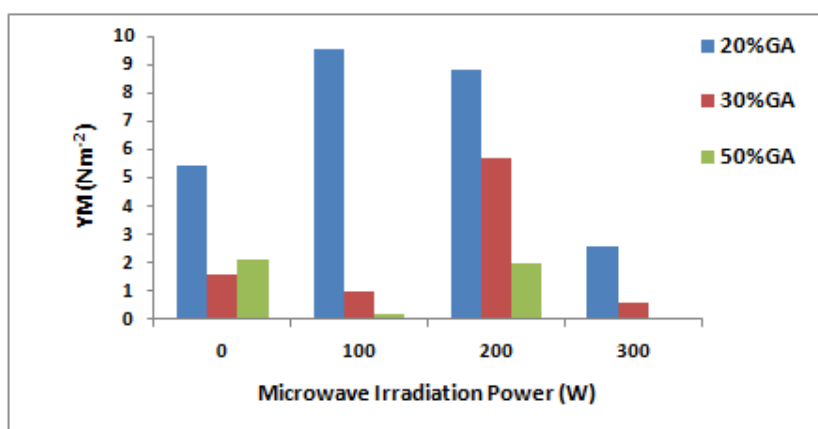


Fig.11: Effect Of Microwave Irradiation Power on Young's Modulus of Gum Arabic Exposed for Three Minutes.

The elongation at break(%), an index of flexibility, of the films for the varying microwave power obtained from the experimental GA concentrations can be seen in Figs 12-14 for 1,2 and 3 minutes of exposure respectively.

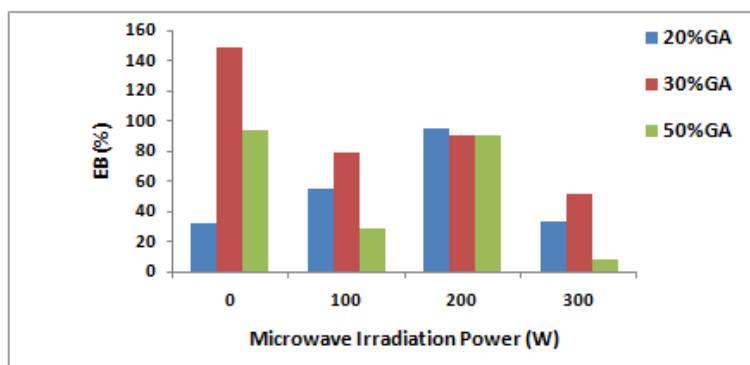


Fig. 12: Effect of Microwave Irradiation Power on Elongation (%) Of Gum Arabic Exposed For One Minute.

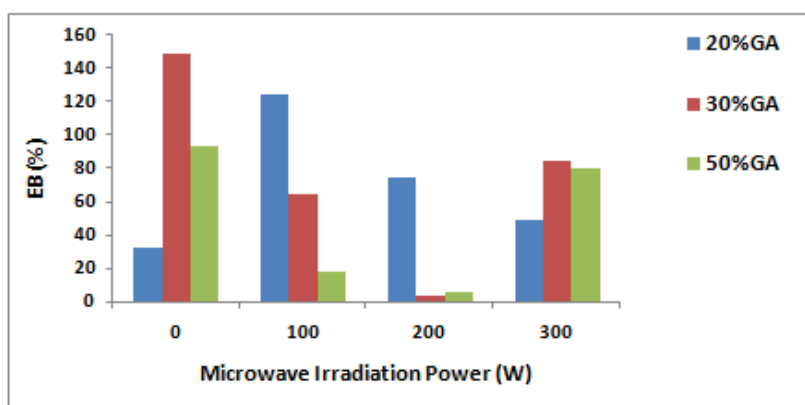


Fig. 13: Effect of Microwave Irradiation Power on Elongation (%) Of Gum Arabic for Two Minutes.

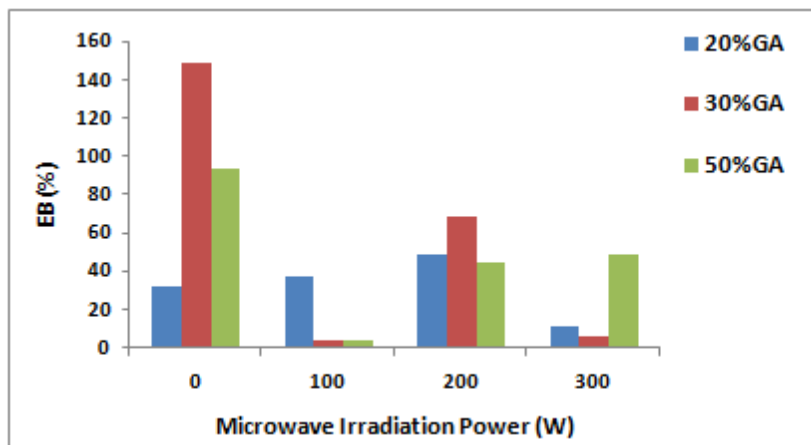


Fig.14: Effect of Microwave Irradiation Power on Elongation at Break (%) Of Gum Arabic for Three Minutes.

Following similar trend, it clear that the film obtained from the unexposed at 30 %GA is more flexible, having EB of 148.5% , but weaker with TS of 3.52Nm⁻², than all the microwaved samples. The most elongated at break for the exposed samples was that of film processed from 20% at 100W for 2 minutes having 124.5% EB which also has the least TS of 3.3Nm⁻². The film from 30% GA microwaved at 300W has the highest value of TS but has one of the least EB (%). These are the usual characteristics of thermoplastics and show that gum Arabic is a thermoplastic biopolymer (Turoti et al, 1999).

Figures 15-17 show that the energy at break(EAB, J), a measure of film toughness, is highest for the film of unexposed 20%GA solution at a value of 5.231J, over all the films obtained from the remaining unexposed and all the ones from exposed GA solutions. However, we consider the overall greater advantages of the other mechanical properties of films obtained from the solutions exposed to microwave irradiations as presented in the previous sections rather than the single marginal advantage of EAB of film from the unexposed 20%GA. In a previous study, Turoti et al., (2015) demonstrated that the presence of some metals e.g. Ti, in some coatings has positive relationship with mechanical properties. Table 4 shows that 0.02g of TiO₂ pigment

present in film processed from 50%GA previously exposed to 300W microwave irradiation for 1 minute could improve most of the mechanical properties of pristine GA including the EAB of the 20%GA above in agreement with an earlier similar works of Alvin et al (2009), Kormin et al (2013), Zhu et al (2003).

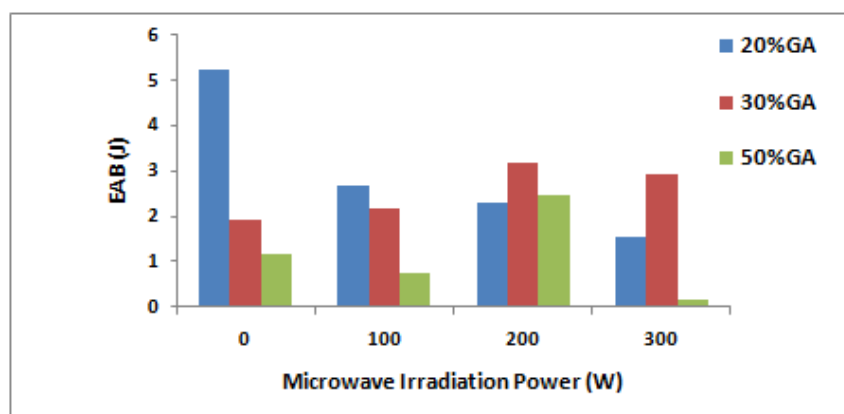


Fig. 15: Effect of Microwave Irradiation Power on Energy at Break (J) Of Gum Arabic for One Minute.

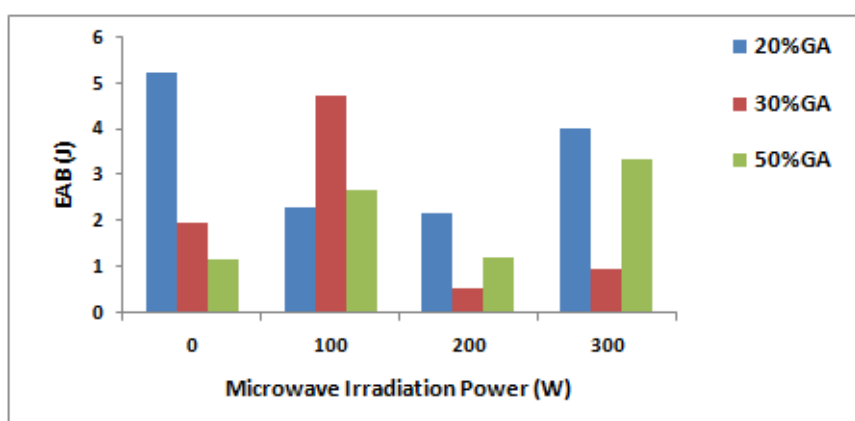


Fig. 16: Effect Of Microwave Irradiation Power On Energy At Break (J) Of Gum Arabic Exposed For Two Minutes.

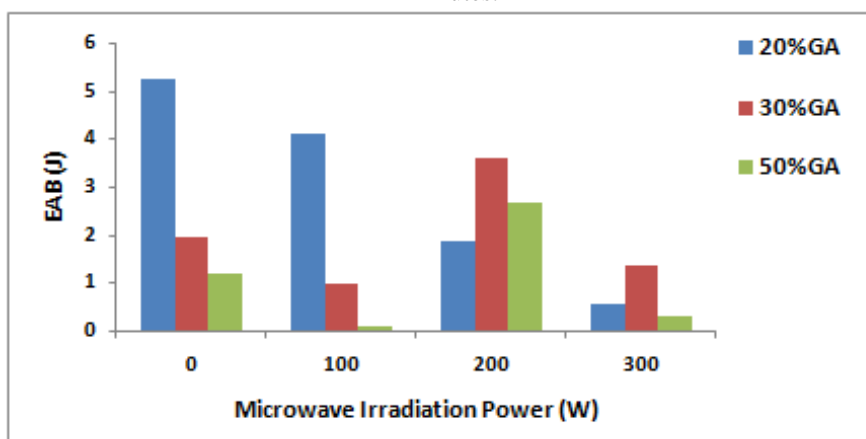


Fig. 17: Effect Of Microwave Irradiation Power On Energy At Break Of Gum Arabic Exposed For Two Minutes.

The results of the mechanical properties show that microwave irradiation subject GA solutions generally to two stages of transformations, namely, separation(disentanglement, softening)of GA chains by microwave heating followed by polar alignment via oscillatory processing/orientation of molecular chains by microwave irradiation. The two stages are more favored at greater microwave power of 300W and higher GA concentrations of 30 and 50% resulting into improved mechanical properties. Thus the GA sample passes

through a softening stage of its glass transition temperature, which occur at lower temperature, and a second crystallization stage at higher temperature, organized by the microwave action on the solution, at shorter and longer exposure time respectively (see Table 3).

Table 3 shows the mechanical properties of films obtained from 50% GA solution exposed to varying microwave powers for 1 minute followed by pigmentation using 0.02g of TiO₂. The pigment was dispersed by mechanical stirrer at the same speed for 100 minutes.

Table3: Mechanical properties of pigmented film obtained from 50% GA solution exposed to varying Microwave Irradiation Powers (W) for 1 minute.

Sample	TSx10 ⁻⁵ (Nm ⁻²)	YM (Nm ⁻²)	EB(%)	EAB x10 ⁻³ (J)
GA alone(unexposed)	4.5	2.1	93.4	1.2
GA+ Pigment(unexposed)	5.1	5.7	118.6	4.5
GA+100W +Pigment	4.3	2.1	211.0	4.6
GA+200W +Pigment	4.6	2.9	158.8	6.0
GA+200W +Pigment	7.6	57.3	102.3	6.5

It is clear that the greater the microwave power the greater the improvement of the TS, YM and EAB while the EB was the least amongst the pigmented samples. The presence of the pigment actually leads to improved flexibility probably by penetration into the polymer chains, and strength probably due to greater metal content and some of the strong hydrogen atoms in this white pigment are known to carry hydroxyl groups which can form strong hydrogen bonds or may even be exchanged with the acidic groups of the glucuronic acid of the gum Arabic chain (Kaluza, 1981)

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IV. Conclusion

From this study on the use of microwave processing for improvement of mechanical properties of the films of the biopolymer, Gum Arabic, a more technological application of the agro waste can be employed. Microwave-processed gum Arabic at 50% concentration in aqueous medium can produce films of tensile strength up to the value of $2.5 \times 10^6 \text{ Nm}^{-2}$, elastic modulus of 57 Nm^{-2} with appropriate level of other improved mechanical properties can be obtained from the readily available, cheap and non-toxic natural product, gum Arabic.

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