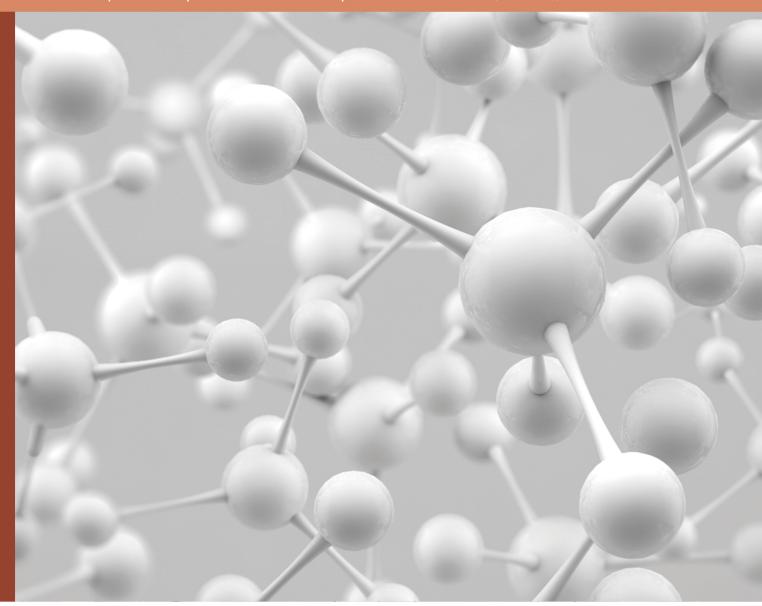


# 02

### Organic Polymer Material Research

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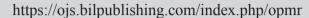
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### ARTICLE

### Allyl Glycidyl Ether-modified Animal Glue Binder for Improved Water Resistance and Bonding Strength in Sand Casting

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### ABSTRACT

This paper aims to develop a modified animal glue sand binder for foundry casting with improved water resistance and bonding strength. An efficient method is reported by using sodium hydroxide as the catalyst to improve the operability of animal glue binder and allyl glycidyl ether as the modifier to improve the water resistance and bonding strength. Sand specimens prepared using allyl glycidyl ether-modified animal glue binder were cured by compressed air at room temperature. The proposed method saves energy and is environmentally friendly and inexpensive. Compared with unmodified animal glue binder, standard dog bone sand specimens with allyl glycidyl ether-modified animal glue binder had higher tensile strength of 2.58 MPa, flowability of 1.95 g, better water resistance (a lower decrease in tensile strength at 25 °C and relative humidity of 60%), and good collapsibility. This allyl glycidyl ether-modified animal glue binder is suitable for practical application in the foundry industry.

### 1. Introduction

Poundries play an important industrial role [1]. A key technical component is the casting binder. Organic and inorganic binders are extensively used in the foundry industry [2-9]. However, pyrolysis of organic substances from these binders emits pollutant gases that are harmful to people and the environment. In comparison with organic binders such as phenolic urethane, inorganic binder such as sodium silicate has low content of organic substances and is recognized as the most environmentally friendly foundry binders [10-12]. But the silica gel has lower strengths after curing, therefore a large ratio of water glass/sand is

required, this would cause lower flowability <sup>[7]</sup>. CO<sub>2</sub> and ester hardened sodium silicate binder has poor collapsibility, and microwave hardened sodium silicate binder has a large hygroscopicity and high requirement of casting mold. To meet the increasing environmental demands, seeking environmentally friendly high-performance binders is still an urgent issue. In recent years, researchers reported the use of natural raw material-based binders derived from plants, such as starch and its derivatives <sup>[13-19]</sup> and plant protein <sup>[20]</sup>. Modified starch can be used as a binder in the foundry industry <sup>[13,21-23]</sup>. NaHSO<sub>3</sub>-modified soy protein had better adhesion performance and water resistance than traditional soy protein isolate adhesive <sup>[20]</sup>.

Animal glue is derived from renewable natural sourc-

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es such as animal's skin, bone, and tendon. Animal glue mainly contains various types of polypeptide molecules. or long chains of amino acids [24]. It is non-toxic, biodegradable, and environmentally friendly; however, it cannot be directly used as a binder in the foundry industry due to its poor mechanical properties, poor water resistance, and low operability. Liu et al [25]. prepared an animal glue binder using acrylic acid, ammonium persulfate, and glucose at a mass ratio of 30:3:15 as a modifier, and obtained a final tensile strength of 3.36 MPa with the addition of 3% binder to sand; however, heat curing at high temperature (180 °C for 20 min) was required, which is highly energy-consuming and may cause oxidative decomposition of the proteins. Liu et al. [26] used glycerin, glycol, and dextrin at a mass ratio of 9:16:15 to modify animal glue, and cured experimental sand samples by blowing high-pressure carbon dioxide gas at a flux of 0.7 m<sup>3</sup>·h<sup>-1</sup> for 60 s. The original and final (24 h) strengths of the standard dog bone sand specimens were 0.7 and 4.2 MPa, respectively. For this binder, the original strength was low, a long time (24 h) was required to reach a suitable tensile strength, and a study of the water resistance of the standard dog bone sand specimens was needed.

The present study aimed to develop a novel modified animal glue binder having advantages of good mechanical properties, good water resistance, low cost, environmentally friendly characteristics, and low energy consumption by using allyl glycidyl ether as the modifier. Allyl glycidyl ether was expected to react with active hydrogens of hydrolyzed animal glue, thus improved its water resistance. Experimental standard dog bone sand specimens were cured by blowing air.

### 2. Materials and Method

Animal glue was obtained from Hubei Jusheng Technology Co. (China). Allyl glycidyl ether (analytical reagent (AR), 99%), NaOH (AR,  $\geq$  96%), and Ca(OH) $_2$  (AR,  $\geq$  95%) were purchased from Aladdin Co. (China). Standard sand (ZGS-50/100(60) grade) was received from Chongqing Changjiang Modeling Materials Group Co. (China).

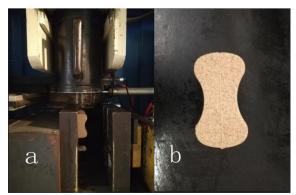
The allyl glycidyl ether-modified animal glue binder was prepared according to previous methods with some modifications <sup>[24,25]</sup>. Animal glue (50 g) and distilled water (45 g) were placed in a 250-mL three-necked flask equipped with a mechanical stirrer. The mixture was kept at room temperature for 10 min until the solution was sufficiently swollen, heated to 40 °C and reacted for 30 min, and then heated to 50 °C and 40 mass% NaOH (5 mL) added dropwise as the catalyst. After hydrolysis for 30 min, allyl glycidyl ether (0.75 g) was added into the

mixture as the modifier and allowed to react for 90 min. The reaction system was cooled to room temperature and approximately 100 g allyl glycidyl ether-modified animal glue binder was obtained as a brown liquid. A similar procedure was used to prepare unmodified animal glue binder that contained no modifier as a control.

Viscosity of the allyl glycidyl ether-modified animal glue binder was measured using an NDJ-4 rotary viscometer (Shanghai Precision Science Instrument Co., China) at H grade, 60 rpm·min<sup>-1</sup>, and 25 °C.

Approximately 1.0 g modified or unmodified animal glue binder was placed in a crucible and dried at 105 °C to constant mass. The resultant solid was ground to powder with a mortar and subjected to elemental, thermogravimetric (TGA), and Fourier transform infrared (FTIR) analyses using an Elementar Vario Micro Select instrument (Germany), TGA209F1 thermogravimetric analyzer (NETZSCH, Germany), and Spectrum 400 spectrometer (Perkin-Elmer, USA) over the 4000–400 cm<sup>-1</sup> region, respectively.

Anhydrous calcium hydroxide (8 g) and standard sand (1000 g) were mixed with stirring for 15s. Allyl glycidyl ether-modified animal glue binder (40 g) was then added and coated sand was obtained by further mixing with stirring for 90s. The uniformly coated sand was placed in the core shooting funnel for a core shooting time of 5s and core shooting pressure of 0.5 MPa. Then the coated sand was placed into the 8-shape mold. The standard dog bone sand specimens was prepared by curing the coated sand using a core shooting machine (OuNai Mechanical Mold Co., China) at a compressed air pressure of 0.5 MPa and blowing time of 5 min. The samples were removed for characterization. All tests were repeated three times and averaged values are reported. Tensile strength of the standard dog bone sand specimens was determined according to AFS Test Procedure 3301-00-S, as per the AFS Mold and Core Test Handbook [27] using an SWY tensile strength testing machine (Chongqing Changjiang Modeling Materials Group Co., China). Figure 1 shows the dog bone specimen and core box.



**Figure 1.** The core box(a) and dog-bone specimen (b)

Flowability of the coated sand was tested according to the Machinery Industry Standards of China (Title: Wet inorganic binder coated sand for nonferrous metal foundry; Standard No: JB/T 13082-2017). The test procedure was described as follows. A hole of diameter 12 mm (height from the center of the cylinder to the bottom of the sample tube was 16 mm) was opened on the side of a 50 mm diameter sample tube (smooth inner wall). Prior to a test, the hole was plugged with a plunger and 185 g of coated sand placed into the sample tube. The plunger was then pulled out and the coated sand hammered 10 times. The combined mass of sand left in the hole and the extruded grit was used to characterize the flowability of the coated sand. Figure 2 shows equipment for measuring the flowability of the coated sand.



**Figure 2.** The equipment for measuring the flowability of the coated sand

Water resistance was evaluated by placing the samples in a constant-temperature and humidity chamber at 25 °C and relative humidity of 60%, then removing them for tensile strength measurements after different times. To determine core collapsibility, the standard dog bone sand specimens were kept at 400, 500, and 600 °C for 5 min and then cooled to room temperature and their tensile strength measured. Gas production was measured at 850 °C using an SFL gas evolution test apparatus (Beijing Jiatian Foundry Material Technology Co., China) using an accurately weighed (1  $\pm$  0.01 g) finely divided sand specimens.

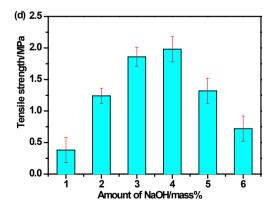
### 3. Results and Discussion

Animal glue is a high-molecular-mass polymer with a

particularly complicated spatial structure in solid state (Figure 3a), so it is not suitable for use as a binder in the foundry industry due to its large intrinsic viscosity and poor fluidity. Animal glue is hydrolyzed to reduce its viscosity using bases, such as NaOH, KOH, and Ca(OH)<sub>2</sub>, as the catalyst. Of these, NaOH was reported to perform best [25]; therefore, NaOH was selected as the catalyst and the effect of NaOH amount on the tensile strength of standard dog bone sand specimens was investigated (Figure 3d). The tensile strength of standard dog bone sand

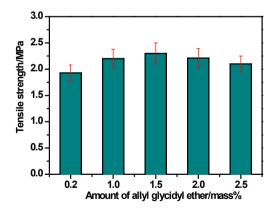
Specimens increased from 0.38 to 1.98 MPa as the NaOH content increased from 0.0 to 4.0 mass%, based on the solid content of animal glue binder. The tensile strength decreased to 1.32 and 0.72 MPa as the NaOH content was further increased to 5.0 mass% and 6.0 mass%, respectively. This indicated that 4.0 mass% of NaOH was the optimum catalyst value. As NaOH was added to raw animal glue, the amide bonds in the glue were progressively hydrolyzed and its spatial structure was destroyed, resulting in a low molecular mass and viscosity. At 4.0 mass% NaOH, the molecular mass and viscosity of the modified animal glue were optimum for its use as a sand binder. Further increase of NaOH caused excess hydrolysis of the glue, resulting in further lowering of the molecular mass and viscosity, which led to decreased tensile strength of the standard dog bone sand specimens.





**Figure 3.** The morphology of raw animal glue (a), unmodified animal glue binder (b), and modified animal glue binder (c). Effect of mass ratio of NaOH on tensile strength of standard dog bone sand specimens (d)

The hydrolyzed animal glue is a brown liquid (Figure 3b) which possesses many active hydrogens, which would cause poor water resistance if directly used as a binder in the foundry industry. Allyl glycidyl ether, selected here as the modifier, was expected to improve water resistance by reacting with the active hydrogens of the hydrolyzed animal glue. Allyl glycidyl ether modified animal glue exhibits a dark brown color (Figure 3c). The effect of allyl glycidyl ether content on the strength of standard dog bone sand specimens was studied. For a modification time of 1.0h, the tensile strength of standard dog bone sand specimens increased from 1.93 to 2.20 MPa as the content of allyl glycidyl ether increased from 0.2 to 1.0 mass% (based on the solid content of animal glue binder), and reached a maximum value of 2.30 MPa at an allyl glycidyl ether content of 1.5 mass%. This demonstrated that 1.5 mass% was the optimum modifier content. Further increase to 2.0 and 2.5 mass% allyl glycidyl ether caused a slight decrease in sample strength to 2.21 and 2.10 MPa, respectively (Figure 4). Because allyl glycidyl ether reacted with the active hydrogens of animal glue, the molecular mass of the binder gradually increased, resulting in improved strength as the allyl glycidyl ether content increased. Maximum strength was obtained when all active hydrogens in the binder were completely replaced; however, when excess ally glycidyl ether was added, hydrolysis and self-polymerization of the modifier occurred, which caused a slight decrease in strength of the standard dog bone sand specimens.



**Figure 4.** Effect of amount of allyl glycidyl ether on tensile strength of standard dog bone sand specimens

The effect of modification time on the viscosity of the animal glue binder was studied (Table 1). As the modification time increased, the viscosity of the binder decreased. This was attributed to hydrolysis of the glue. When the modification time was less than 1.5 h, the tensile strength of standard dog bone sand specimens increased as the

modification time increased, reaching a maximum value of 2.58 MPa at a modification time of 1.5h. Further increase in modification time caused a decrease in the tensile strength of the standard dog bone sand specimens. Flowability of the standard dog bone sand specimens increased with the modification time due to decreased viscosity of the modified binder. When the modification time was short, the viscosity of the binder was too large and unsuitable for binding sand, so the tensile strength and flowability of the standard dog bone sand specimens were low. As the modification time increased, the animal glue was further hydrolyzed, accompanied by a decrease in viscosity: the modifier reacted with the glue, resulting in an increase in tensile strength and flowability of the standard dog bone sand specimens. An optimum tensile strength of 2.58 MPa was obtained at a modification time of 1.5h, when flowability of the samples was 1.95g. When the modification time exceeded 1.5h, the viscosity of the binder was too low, causing a decrease in sand strength.

**Table 1.** Effect of modification time on properties of modified animal glue binder and standard dog bone sand specimens

Modification time (h)	Intrinsic viscosity of modified animal glue binder (×10³ mPa·s)	Tensile strength of standard dog bone sand specimes (MPa)	Flowability of stan- dard dog bone sand specimens (g)
0.5	3.40±0.15	2.21±0.10	1.80±0.20
1.0	1.95±0.15	2.28±0.15	1.88±0.20
1.5	1.40±0.10	2.58±0.20	1.95±0.20
2.0	1.04±0.10	2.16±0.18	2.23±0.30
2.5	0.85±0.10	2.05±0.15	2.45±0.30

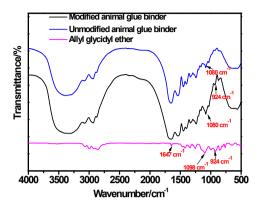
Elemental analyses of unmodified and modified animal binder are listed in Table 2. Compared with unmodified animal glue binder, the modified binder had slightly lower N and S contents and higher C and H contents due to the addition of allyl glycidyl ether.

**Table 2.** Elemental compositions of unmodified and modified animal glue binders

Animal glue	N (%)	C (%)	H (%)	S (%)	C/N ratio	C/H ratio
Unmodified animal glue	15.84	45.05	6.37	0.43	2.85	7.07
Allyl glycidyl ether modified animal glue	15.54	45.25	6.59	0.39	2.91	6.87

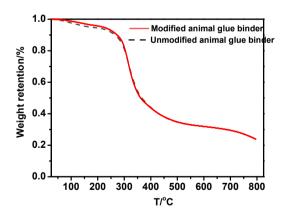
Figure 5 shows the FTIR spectra of unmodified animal glue binder, allyl glycidyl ether-modified animal glue binder, and allyl glycidyl ether. The appearance of an absorption band at 924 cm<sup>-1</sup> and the increase in absorbance intensity at 1080 cm<sup>-1</sup> for the modified binder illustrates

that allyl glycidyl ether reacted with hydrolyzed animal glue [28].



**Figure 5.** Fourier-transform infrared spectra of unmodified animal glue binder, allyl glycidyl ether-modified animal glue binder, and allyl glycidyl ether

Thermogravimetric curves of the modified and unmodified animal glue binders are presented in Figure 6. The mass loss below 200 °C was assigned to the removal of adsorbed water; that between 250 and 450 °C was attributed to the dehydration of macromolecular substances and loss of bound water in protein molecules. Compared with unmodified animal glue binder, the modified binder had a slightly lower mass loss below 300 °C, which indicated that it had slightly better stability.



**Figure 6.** Thermogravimetric curves of unmodified and modified animal glue binders

Collapsibility of the mold has an important impact on the quality of a casting. Standard dog bone sand specimens that had been cured by blowing air were heated to 400, 500, and 600 °C for 5 min, cooled to room temperature, and then analyzed for residual tensile strength.

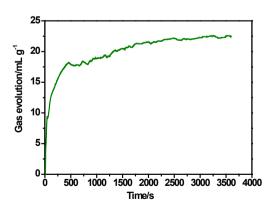
As shown in Table 3, residual strength of the standard dog bone sand specimens decreased as the temperature increased: residual tensile strengths of standard dog bone sand specimens that were heat treated at 400, 500, and 600 °C for 5 min were 0.71, 0.10, and 0.00 MPa, respectively. The residual tensile strength of the sample heated at 500 °C for 5 min was close to 0.00 MPa. For comparison, for an animal glue binder modified with acrylic acid, ammonium persulfate, and glucose at a mass ratio of 30:3:15, residual tensile strengths of standard dog bone sand specimens heat treated at 500, 600, and 700 °C for 10 min were 0.39, 0.08, 0.00 MPa, respectively [21]. This indicated that the standard dog bone sand specimens using the allyl glycidyl ether-modified animal glue binder had good collapsibility. The mold could be easily removed on completion of casting and there was no residual grit on the device, thus producing a cast product with good quality.

**Table 3.** Residual tensile strength of standard dog bone sand specimens heat treated at different temperatures

Temperature (°C)	Residual tensile strength (MPa) <sup>a</sup>
400	0.71
500	0.10
600	0.00

*Note:* <sup>a</sup> Residual tensile strength was determined after cooling the standard dog bone sand specimens to room temperature.

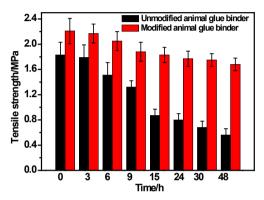
The gas production of sand samples is an important indicator for the quality of casting. A lower value implies better quality because such samples have fewer defects and stomata. Figure 7 showed the gas evolution of sand samples with the allyl glycidyl ether-modified animal glue binder. Gas evolution was approximately 22.5 mL·g $^{-1}$  at 850 °C at 3600 s.



**Figure 7.** Gas evolution of sand sample with allyl glycidyl ether-modified animal glue binder

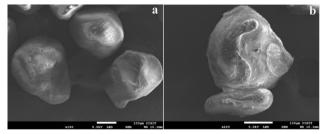
The water resistances of standard dog bone sand specimens were measured at 25 °C and relative humidity of

60%. As shown in Figure 8, samples prepared using the allyl glycidyl ether-modified animal glue binder had better water resistance than those using the unmodified binder. After 48 h, tensile strength of samples using the modified binder slightly reduced from an initial strength of 2.21 to 1.7 MPa; however, the corresponding tensile strength of samples using the unmodified binder significantly reduced from 1.83 to 0.56 MPa. The improved water resistance of samples using the modified binder was attributed to the interaction of allyl glycidyl ether with the active hydrogens of the animal glue [23].



**Figure 8.** Water resistance of modified and unmodified animal glue binders

The bonding effect of the modified animal glue binder with quartz sand gravel is shown in the scanning electron micrographs in Figure 9. Native quartz sands had a smooth surface, and the gravels did not bind together (Figure 9a). After coating with the allyl glycidyl ether-modified animal glue binder, the surfaces of the sands were coated by the binder and became slightly rougher (Figure 9b). The grafted animal glue binder was uniformly dispersed in the gaps between the particles and bound them together, thus improving the sand strength.



**Figure 9.** Scanning electron micrographs of (a) native quartz sand gravel and (b) quartz sand gravel coated with allyl glycidyl ether-modified animal glue binder

### **Conclusions**

Modified animal glue binder was efficiently prepared

using NaOH as the catalyst and allyl glycidyl ether as the modifier. The modification conditions were optimized using 4.0 mass% NaOH and 1.5 mass% modified animal glue binder, based on the solid content of animal glue binder, and a modification time of 1.5h. Using this modified animal glue as a casting binder only required a low-energy, environmentally friendly, and inexpensive curing procedure using compressed air at room temperature. The modified animal glue binder exhibited improved water resistance, good binding ability, low gas evolution, and appropriate collapsibility. The improved water resistance was ascribed the reaction of the modifier, allyl glycidyl ether, with the active hydrogen of the hydrolyzed animal glue. It can be used as a casting binder in the foundry industry.

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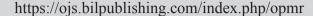
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### **REVIEW**

## Mini Review: Chemical Mesoscopics Notions in the Explanation of Polymeric Materials Modification Mechanism with Participation of Metal Carbon Mesocomposites

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### **ABSTRACT**

The paper is dedicated to the consideration of the chemical mesoscopics notions application for the explanation of polymeric materials modification mechanism by the metal carbon mesoscopic composites. The main peculiarities of these nanosized particles are following: a) the presence of unpaired electrons on the carbon cover; b) the structure of carbon cover consists from poly acetylene and carbine fragments; c) the atomic magnetic moment of inner metal is equaled to more than 1,3  $\mu B$ . The metal carbon mesocomposites activity depends on the medium and conditions influence because of the possible changes of the phase coherency and quantization of negative charges.

### 1. Introduction

he article is presented as the review of series of papers, manuscripts and patents on the obtaining, investigations and applications of uncials mag-

netic mesoparticles which are mesoscopic metal carbon composites [1-25]. The mesoscopic composites can be participates in reactions, especially, in radical processes and reduction oxidation processes. This activity may be used in modification processes mesocomposites accompanied

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by the magnetic characteristics changes in modified mesoscopic composites. The expansion of metal carbon mesocomposites application chances takes place.

### 2. Discussion of the Polymeric Materials Modification Mechanism by Mesocomposites (on the Base of Series Experimental Results)

The mesocomposites activity in the different media (materials) is changed in the dependence on polarity or polarization of their media. Therefore the modification conditions for the different materials can be differed from each other. It's noted,<sup>[1,2]</sup> that the mechanism of polymer modification by the nanostructures or mesoparticles differs from the correspondent mechanism at the micro particles using. The nanostructures radiate the quants of negative charges which increase polarization of medium and lead to the self organization of medium molecules accompanied the density growth.

According to scheme (Figure 1), the possible polarization leads to the increasing of medium (material) density owing to the regular orientation of material fragments or the macromolecules self organization with the creation of super molecular and crystalline structures. On the scheme the direct motion of electron quants shows by the arrows, and the polar (functional) groups - by the correspondent signs  $\mathcal{Q}$ , macromolecular chain of polymer is designated as line, the presence of mesoscopic composite is noted as MC.

The polarization extent depends on the quants electromagnetic radiation phase velocity. This velocity depends on the medium polarity and can be decreased at the polarity fall.

Figure 1. Scheme of polarization at charge quantization with expansion of quant influence on materials polar groups

The polarization increasing of polymer macromolecules at the mesoscopic composite action can be expressed by the following equation:

$$P_{com} = \sum p_{fg} + p_{NC}, \qquad (1)$$

where  $P_{com}$  - the common (summary) polarization,  $\Sigma p_{fg}$  - sum of functional groups polarizations,  $p_{NC}$  - the polarization (or dipole moment) of mesocomposite

The electromagnetic radiation phase velocity will be decreased in the media with high dielectric constant according to following formula:

$$v = c/\sqrt{\epsilon}$$
, (2)

where v - the phase velocity of electromagnetic radiation, c - the light velocity,  $\epsilon$  - dielectric constant.

When the dielectric constant is increased the decrease of mesocomposite influence on the media arises and the self organization process is finished.

Depending on the development of self organization process (single measured - 1D, double measured - 2D, third measured - 3D) the super molecular structures (mesoparticles) of correspondent forms and sizes are organized. The surface energy of embryos increased influences on the mesoparticles formation. This energy can be express as the sum of energetic parts for the realization of different movements:

$$E_{S} = E_{(tr)} + E_{(r)} + E_{(osc)} + E_{(em)}.$$
 (3)

where  $E_s$  - surface energy of macromolecule (mesoparticle),  $E_{(tr)}$  - part of translational motion energy,  $E_{(r)}$  - part of rotary motion energy,  $E_{(osc)}$  - part of oscillatory motion energy,  $E_{(em)}$  - part of electron motion in surface layer.

In accordance with the formation of mesoparticles which have the identical orientation with each other, the parts of translational motion energy and of rotary motion energy will be near to zero. Therefore the main contribution into the mesoparticle surface energy will be bringing the oscillatory motion and transport of electrons in surface layer of macromolecules (mesoparticles). Then the change of character of quants radiation wave propagation from 2D (in the surface plane) to 3D (in the space field at surface).

The composition polarization is possible because of the presence of the charge quantization with the wave expansion on polar functional groups of media (for example, polymer macromolecule, Figure 1). In table 1 the instance of fine dispersed suspension Cu-C mesocomposite (hardener for epoxy resin) is shown. According to data of Table 1, the decreasing of mesocomposite quantity to 0,001% leads to the intensity increasing of some fields in IR spectra.

**Table 1.** The change of peaks intensity depending on Cu-C mesocomposite concentration

N	cm <sup>-1</sup>	$I_1/I_0$	$I_{0,01}/I_{0}$	$I_{0,001}/I_{0}$	Atomic groups
1	1050	1,235	1,411	1,686	C-O-C st
2	1450	1,179	1,590	1,744	С-Н
3	1776	1,458	1,347	1,691	C=O st as
4	1884	1,463	1,412	1,678	C=O st sy
5	2860-3090	1,182	1,545	1,750	С-Н

At the second day of that suspension existence the

floccules are formed and peaks intensity sharply drops. However the suspension activity can be increased with the using of ultrasound treatment (Table 2). The treatment optimal duration determined as 7 minutes (the peak intensity in IR spectra is increased in 2-4 times).

**Table 2.** The changes of peaks intensity in IR spectrum of Cu-C mesocomposite depending on the duration of ultrasound treatment

v(cm <sup>-1</sup> )	$I_7/I_0$	$I_{10}/I_{0}$	Atomic groups
1776,6	3,7932	0,7575	C=O st as
1844,1	2,5065	0,9115	C=O st sy
3039,1	2,3849	0,9589	С-Н

The charge (electron) quantization should lead to the macromolecule electron structure change and, as corollary, to change sub molecular structures of polymeric substances. Therefore the special film of nanostructured materials, for example, polyvinyl alcohol, polymethyl metacrylate, polycarbonate, which contain metal carbon mesocomposite in the minute quantities (10<sup>-1</sup> - 10<sup>-5</sup> %) are prepared. The films obtained are studied by x-ray PES and by AFM. The investigations by x-ray PES show that the films based on polycarbonate have more changes of electron structure at the minute quantities introduction of Copper Carbon mesocomposite in comparison with other polymeric films because these films are more polarized.

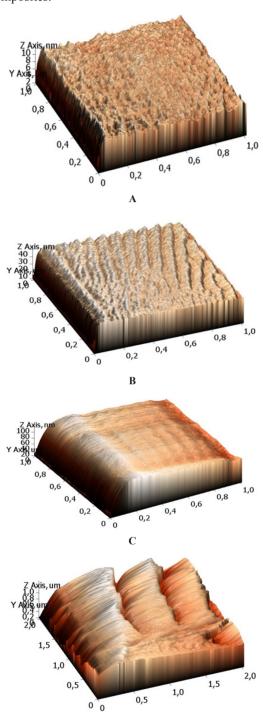
According to the results of C1s spectra for polycarbonate, contained the different minute quantities of Cu-C mesocomposite, can note that after concentration equaled to  $10^{-2}$  % of Cu/C mesocomposite the peaks correspondent to sp<sup>2</sup> and sp<sup>3</sup> peaks are appeared in these spectra. In other words, the "stamp" of mesocomposite which is used during modification is appeared.

That "stamp" is observed also at the mesocomposite containing, equaled to  $10^{-5}\%$ , in polycarbonate film. It's noted, that the relation between sp² and sp³ peaks changes. For instance, the intensity of sp² hybridization carbon peak is upper the intensity of sp³ peak in the concentration interval from 0,01 to 0,001% of mesocomposite. The change of concentration to  $10^{-4}$  % bring the proximity of intensities sp² and sp³ peaks.

For the decision of question about the nanostructure influence on sub molecular composition structures the atomic force microscopy method is applied. Below some images of polycarbonate nanostructured films surface are presented. Polycarbonate is modified by Cu-C mesocomposite minute quantities (from 10<sup>-1</sup> to 10<sup>-4</sup> %). As follow from AFM images (Figure 2), the surface layers structure strongly changes at the concentration of Cu-C MC equaled to 10<sup>-4</sup> % the transition from two-dimensional level to three-dimensional

level of sub-molecular structures orientation. This fact is confirmed by the growth of sp<sup>3</sup> in comparison with sp<sup>2</sup> peak from x-ray photoelectron C1s spectra.

The changes of the image surface are also observed for the poly methyl metacrylate films modified by Cu C mesocomposites.



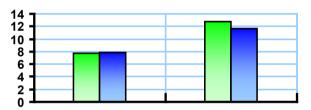
**Figure 2.** AFM images of polycarbonate nanostructured films surface: A - 0,1% Cu-C MC; B - 0,01% Cu-C MC; C - 0,001% Cu-C MC; D - 0,0001% Cu-C MC

It's necessary to note that the results obtained by AFM conform by the x-ray PES data (on C1s spectra).

Thus, the self organization mechanism for polymeric compositions modified by the metal carbon mesocomposite minute quantities is concluded in the conditions creation for composition polarization, which leads to the great change of electron and sub molecular structures of materials. Certainly, these changes influence on the modified materials properties.

Below in the example, the epoxy compositions with different additives, which include mesoscopic composites, are investigated. As cross-linking agent is used the fine dispersed suspension on based of isomethyl tetra hydro phthalates anhydrate and Copper Carbon mesocomposite.

On the Figure 3 the results of modification epoxy compounds (materials 1 and 2) are given. The process of modification by Copper Carbon mesocomposite in quantity equaled to 0,005% improves the adhesion characteristics for the material 1 (green) on 59,77% and for the material 2 (dark blue) on 47,17%.



**Figure 3.** Comparison of adhesion strength for materials 1 (green) and 2 (dark blue) before (A) and after (B) the modification by Copper Carbon mesocomposites

It's possible also other applications that nanostructures owing to its uncial structure and properties.

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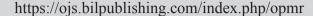
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### **REVIEW**

### **Physicochemical Properties of Gum Arabic**

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### ABSTRACT

Gum Arabic is harvested commercially from wild trees throughout the Sahel from Senegal and Sudan to Somalia and in the northern part of Nigeria. Clumps of gum Arabic were collected and dirt particles were removed, the samples were dried and grounded to fine powder. The Physicochemical properties of gum Arabic was determined using standard methods and the values obtained showed; Moisture content  $6.9\% \pm 5$ , Soluble content 90.6%, Viscosity  $5.45 \pm 3$ Ns/m2, Ash content  $3.2 \pm 4\%$ , Zinc 3mg/kg, Iron 41mg/kg  $\pm 5$ , Manganese 48.2mg/kg  $\pm 5$  and Copper 33.3mg/kg  $\pm 2$ . The gum also contains carbohydrate 0.3ppm  $\pm 2$ , protein 0.75ppm  $\pm 2$ , starch  $0.0076 \pm 10$  and nitrogen 0.12ppm  $\pm 5$ . It was observed that the gum does not contain cadmium and nickel.

### 1. Introduction

cacia Senegal, popularly known as gum Arabic is a leguminous tree crop which belongs to the family Mimisaceae. This family contains over 300 known species including Acacia Senegal and Acacia Seyal and are the most commercially exploited species. Both species are considered as the important economic Acacia plant in the Sudan-Ssahelian ecological zones covering not less than eleven states in the northern part of Nigeria. The species produces the best grade of gum in commercial quantity in Nigeria and it is an important source of foreign exchange earnings [1].

Acacia gum is the dried exudates obtained by tapping the stems and branches of acacia Senegal or other related species. The gum has various uses in the food, textiles, prints and pharmaceutical industries in the world. The trees are planted in shelterbelt to check wind and soil erosion, hence it is used to fight against desert encroachment <sup>[2,3]</sup>. The plant adds nitrogen to the soil through its nitrogen fixing ability and leaf litter fall thereby improving soil fertility <sup>[4]</sup>. Its pods, seeds and leaves are excellent browse and folder for livestock <sup>[5]</sup>.

Gum Arabic is harvested commercially from wild trees throughout the Sahel from Senegal and Sudan to Somalia, although it has been historically cultivated in Arabia and West Asia. Gum Arabic is a complex mixture of polysaccharides and glycoproteins that is used primarily in the food industry as a stabilizer. It is edible and is a key ingredient in traditional lithography and used in printing, paint production, glue, cosmetics and various industrial applications, including viscosity control in inks and in textile industries, although less expensive materials compete with it for many of these roles [6,7].

Gum Arabic is unique among the natural hydrocolloid because of its extremely high solubility in water. Most

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common gums cannot be dissolved in water at concentration higher than about 5% because of their very high viscosities. Gum Arabic is insoluble in oils and in most organic solvents. Whereas most gums form highly viscous solution at low concentration of about 1-5%, high viscosities are not obtained with gum Arabic until concentration of about 40-50% is obtained.

Technically, gum Arabic is classed in a group of substances called arabinogalactan protein. Eighteen different amino acids have been identified in acacia Senegal, although only four comprises more than 10% of the protein. The gum also comprises essentially a complex polysaccharide, consisting mostly of galactose, arabinose, rhamnose and glucoronic acid <sup>[8,9]</sup>. Gum Arabic is useful as hydrocolloid, emulsifier, texturizer and film-former <sup>[10]</sup>.

Substances frequently called gums are hydrocarbons of high molecular mass; others are petroleum products, rubber latex, synthetic polymeric gums, balms and resins. Recently, the term "gum" as technically employed in the industry, refers to plant or microbial polysaccharides and their derivatives that are capable of forming dispersions in cold or hot water, producing viscous mixtures or solutions [11].

### 2. Materials and Methods

### 2.1 Sampling

The sample was harvested from Acacia Senegal. The stem of the specie was cut and the gum oozed out and dried at room temperature of 30°C±5 to form clumps. The clumps were collected and particles such as sand, sticks and dirt were removed from the clumps and preserved with formaldehyde to avoid micro organism infestation which may cause biodegradation of the gum. The samples were dried for easy grounding. The dried samples were grounded to fine power and kept in an air-tight container.

Materials used include; Ethanol, tetraoxosulphate (VI) acid, toluene, anthrone reagent, hydrochloric acid, sodium carbonate, alkaline sodium phenate, sodium hypochlorite, sodium potassium tartrate, dry ammonium sulphate and sodium sulphate. They were supplied and used as standard analytical reagents.

### 2.2 Methods

Metal contents was determined using the Atomic Absorption Spectrophotometer (AAS), Sugar contents and Carbohydrate contents were determined using Anthrone method, Viscosity was determined using Oswald Viscometer, pH was determined using the Jenway 3510 pH meter. Other properties such as Ash content, Moisture content,

Insoluble matter, Soluble matter, Nitrogen and Protein were determined using standard methods.

### 3. Results and Discussion

### 3.1 Results

Table 1. Results of physio-chemical properties

PROPERTIES	VALUES
Ash Content	3.23%
Moisture Content	6.9%
Insoluble Matter	9.4%
Soluble Matter	90.6%
Viscosity	$5.45 Ns/m^2$
pH	8.16

Table 2. Results of Organic Parameters

PARAMETERS	VALUES
Carbohydrate	0.275ppm
Sugar	0.0081ppm
Nitrogen	0.1200ppm
Protein	0.7500ppm

Table 3. Results of Metal Contents Analysis

METALS	VALUES (mg/kg)
Iron	41.0
Manganese	48.2
Copper	31.3
Zinc	3.0
Cadmium	ND
Nickel	ND
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Note: ND means "Not Detected".

### 3.2 Discussion

The results of the physio-chemical properties of gum Arabic (from acacia Senegal) are displayed in Table I. It shows ash content, 3.23%, moisture content, 6.9%, insoluble matter, 9.4% soluble matter, 90.6%, viscosity, 5.45Ns/m² and pH, 8.16. Gum Arabic tends to be more viscous than that of cashew tree exudates. The viscosity of cashew tree exudate is 3.64Ns/m². The reason is that gum Arabic has less moisture contents of 6.9%, compared to that of cashew gum which has a moisture content of 9%. This suggests that acacia tree tends to store much moisture on its body for survival, unlike cashew tree.

From the standard in literature review by Willy Be-

necke, the ash content in gum Arabic is 4%. This has a close value with the result from this research, with a difference of 0.8% approximately. The standard insoluble matter is 1% which is not too close to the result of insoluble matter, 9.4%. The reason could be that the actual specie of gum Arabic used for the standard was not specified because physiochemical properties vary from specie to specie.

Table II displays the content of the organic nutrient in the sample; gum Arabic. It reveals that carbohydrate 0.275ppm, sugar content 0:00816ppm, Nitrogen 0.1200ppm and protein content is 0.7500ppm. Organic parameters seems to be more in cashew tree exudates which has carbohydrate content as 0.5674ppm, protein 1.0200ppm, nitrogen content 0.1640 and sugar content 0.6280ppm. The reason is traced to the different regional areas of their habitation. Nutrients are easily lost to the environment especially in savanna region where acacia trees inhabits. These values give the gum a perfect option to emulsify.

The results from the metal content analysis is given in Table III, which shows iron content; 40mg/kg, manganese; 48.2mg/kg, copper; 31.3mg/kg and Zinc; 3.0mg/kg. Cadmium and nickel was found to be absent. The absence of these two poisonous metals makes the gum applicable in the food industry.

### 4. Conclusion

Gum Arabic is unique among the natural hydrocolloids. Gum Arabic's mixture of saccharides and glycoproteins gives it the properties of a glue and binder which is edible by humans. For artists, it is the traditional binder used in water colour paint, in photography for gum printing and as a binder in pyrotechnic compositions. It has been investigated for use in intestinal dialysis. Pharmaceuticals and cosmetics also use the gum as a binder, emulsifying agent and suspending or viscosity increasing agent [6]. These applications are successfully achieved on the basis of the physicochemical properties which have been established by this research work. The gum is highly viscous and slightly basic and contains little carbohydrate and sugar

compared to protein and nitrogen content, suggesting it to be a leguminous plant. Manganese, zinc, iron and copper are the analyzed trace metal content. These trace metals are vital to the proper growth and development of the acacia plant [7].

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### ARTICLE

### An Insight into the Polymeric Structures in Asian Palmyra Palm (*Borassus flabellifer Linn*)

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### ABSTRACT

Palmyra Palm (Borassus flabellifer Linn) is a native tree with various ecological, medicinal, economic, and sociological benefits from Asian countries. Palmyra Palm tree-based self-reliant lifestyle and Eco-friendly community living which leads to sustainable development can be called palmyra culture. For each component, Palmyra Palm is the most beneficial species that has economic and medicinal value that could sustain adverse climatic conditions and resist natural calamities. Non-edible, edible, and value add-based uses could be widely categorized into the utility of the plant. Palmyra palm tree is working like a non-stop biochemical factory, creates sugars and many otheruseful chemicals. It also produces many useful polymeric compounds such as pectin, cellulose, hemicelluloses, pentosanpolysulfate, polyphenols, and lignin. The current review gives an insight into the structural, biological, and polymeric elements of Asian palmyra.

### 1. Introduction

almyra Palm, Borassus flabellifer Linn (Family: Arecaceae) is the most economically important species, mainly distributed in Asian countries such as India, Sri Lanka, Bangladesh, Myanmar, Indonesia, Thailand, and particularly in dry African tropical regions<sup>[1]</sup>. It is also the official tree of the Government of Tamil Nadu. Asian Palmyra Palm leaves, fruit, and pit are known to provide a livelihood for many small-scale

industries in the Asian region for their economic uses <sup>[2]</sup>. Palmyra Palm is a long-lived tree that can live more than 100 years with a canopy of leaves and a large trunk <sup>[3]</sup>. Each toddy palm tree can endure 6-12 bunches of about 50 fruits per year, 350 fruits are an average crop. In many traditional food items, pulp obtained from ripe fruit is used to cure various diseases of folk medicine <sup>[4]</sup>. Palmyra palm has important non-food applications, such as marketable mats, oils, baskets, timbers, dyes, and fibers <sup>[5]</sup>. The treatment of contaminated water as an

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absorbent of complex hexavalent and trivalent chromium compounds [6], the potential treatment of heartburn, dermatitis, respiratory problems, and diarrhea in traditional medicine [7]. Palmyra Palm trees play an important role in agriculture in the countries of Asia. Palmyra Palm treebased self-reliant lifestyle and Eco-friendly community living which leads to sustainable development can be called palmyra culture. Asian palm sugar is known by other names such as palm jaggery, palm jaggery, Neera, and Gur. It has a unique taste and produces less energy than cane sugar [8]. The sap is used for wine, palm jaggery, and palm sugar, all rich in vitamins and minerals [9-<sup>10]</sup>. Several pharmacological advantages of Asian palmyra have also been reported in traditional medicine, such as antidiabetic [11], analgesic [12], anti-inflammatory [13], wound healing [14], antioxidant [15].

Although the Asian palmyra is widely used, it is reported to be underused and left to spoil most of the fruits, sap, and other parts of the tree. Recovery, reduction, and recovery of post-harvest losses may maintain sustainable agricultural practices with minimal impact on the environment and improve food security [16]. Due to the once widespread use of most of its parts, such as the trunk, foliage, husk, nut, and flesh, Palmyra palm is a "miracle" plant. For furniture and handicraft products, the trunk can be used. To make some crafts, dried leaves and the flexible sticks in the front are woven. To brew wine and vinegar and to make sugar, palm nectar is used. Edible is the flesh inside the nut. The husk or mesocarp can be extracted from a natural food coloring substance. Palmyra palm tree is working like a biochemical factory and creates sugars and thousand of chemicals. Among them, many are polymers such as pectin, Cellulose, Hemicelluloses, pentosanpolysulfates, polyphenols, and lignin. The present review focuses on the reporting of the biological and polymeric components contained in the Asian Palmyra Palm.





**Figure 1.** Young, matured Asian Palmyra tree and collected Palmyra fruits for the plantation of 4000 seeds at Panaiyaanmai (Palmyra culture)-The centre for self-reliance and sustainable development, Kadayam, Tamil Nadu

### 2. Main Components of Palmyra Palm

Pectin is found in plant tissue in the intercellular or middle lamella region [17]. Pectin, also known as water-soluble fiber, is a polysaccharide. It is employed as a gelling agent and functional food in both the food and pharmaceutical industry. In multiple food processing areas such as jam, jellies and marmalade, low sugar and calorie foods, bakery, further processed meats, and acidified milk drinks, pectin is used in which gelling and viscosification are significant physicochemical properties [18]. Palm fruit provides an inexpensive and environmentally friendly raw material for the extraction of pectin. The economical manner and yield pectin with unique properties of gelling, viscosification, and emulsification. The increasing interest in the Palmyra palm is due to the increasing awareness of the unexploited potential of this tropical resource [5]. It is reported that the Palmyra palm meat obtained from the young inflorescence was found to have a high potential pectin source [19]. The pectin extraction and their yield levels using both ripened and young Asian Palmyra palm sugar meat by altering the microwave irradiation were reported [20]. Palmyra palm fruits have great potential for the pectin sector as an alternative, commercially viable source of pectin. Depending on the extraction conditions, the identified differences in structural and functional properties provide the basis for the extraction.

**Figure 2.** Structures of A) Pectin, B) Galactomannan, and C) Pentosanpolysulfate

Palmyra palm, limited in fat and protein, is a good source of carbohydrates, calcium, magnesium, iron, and fiber. A fibrous kernel is progressively hardened and developed by the tender of Asian palmyra palm pulp. The presence of galactomannan from the soft palmyra kernel was reported [21] and showed that it consisted of mannose and galactose. It is reported that the free radical scavenging effects of carotenoids from Asian palmyra palm pulp and the nature of free sugars and polysaccharides present in the kernel [22].

As it is obtained from renewable resources and is the most abundant biopolymer on the planet, cellulose has become an important polymeric material [23]. Paper is a sheet consisting of cellulosic fibers that are normally produced using mechanical or chemical processes to separate wood cells. Subsequently, the isolated fibers are re-arranged and randomly distributed into a sheet-like framework. The cellulose pulp from hardwood and softwood is usually obtained by the pulp and paper industries. However, an insufficient supply of wood for growing demand has caused industries, such as non-wood fiber plants, to search for alternative fiber sources. A further fibrous source used as a raw material for pulp and papermaking has been the mesocarp of Palmyra palm fruits. There seem to be fewer references in the scientific literature to the pulping of palmyra palm fruit fiber.

Figure 3. Structure of A) Cellulose and B) Hemicellulose [34]

Palmyra palm fruit fibers have the characteristics to be used for papermaking as an alternative raw material for cellulosic pulps. The chemical composition of the strands of palmyra palm fruit fiber from palmyra palm fruits such as holocellulose, cellulose, pentosan sulfate, lignin, and extractives was examined [24] and it was found that the mechanical and physical properties of the acid and alkaline pulps were verified to be of acceptable paper-making quality.

An effective alternative source of cellulose for paper

pulp production is the mesocarp of Palmyra palm fruits. Their findings suggested that the fibers of Palmyra palm fruit are an effective alternative raw material for paper pulp. It offers acceptable properties for pulp and paper sheets and thus makes it possible to profitably exploit agricultural residues from major economic activity.

The natural Palmyra palm fruit fiber cellulose microfibers contain cellulosic semisolid flush that is armored by the fibers. The fibers of ripened Palmyra palm fruits, which are inexpensive, abundantly available, and environmentally friendly, have been extracted from the waste, and it is, therefore, essential to explore their potential usefulness to the technical world. The yield of cellulose in Palmyra palm fruit fibers was found to be more than 50 percent [25].

It is promising to utilize Palmyra palm fruit fiber as the primary source of cellulose. A campaign of studies that extracted cellulose microfibers could prove to be a useful alternative raw material for biopolymer composites, biofuel and cellulose derivatives, and paper pulp applications. An increase in the alpha-cellulose content and a decrease in the lignin and hemicellulose content of cellulose microfibers over raw fibers is shown in the chemical analysis of palmyra palm fruit. For cellulose microfibers extracted from Palmyra palm, an easy and cost-effectively feasible way will further assist in determining appropriate end uses for these fibers and microfibers, which will add value to the harvest [23].

Hemi-cellulose is a cellulose-related polysaccharide. But unlike cellulose, hemicellulose, in addition to glucose, is derived from several sugars. In comparison to a cellulose chain, it consists of a shorter chain [23]. The main hemicellulose in hardwoods consists of compounds that contain glucuronoxylan compounds.

Two saponins in the fruit pulp and tuber flour were identified [26]: steroid spirostmonoglucoside and monorhamnoside-5en-3fi-o11 (25R). Palmyra (Borassusflabellfer L) fruit pulp's main bitter principle has been tentatively identified as a tetraglycoside (flabelliferin II) steroidal saponin containing two glucose and two residues of rhamnose. By the action of naringinase on both crude bitter extracts (containing flabelliferin I and II) and natural fruit pulp, bitterness can be removed. To produce other flabelliferins, naringinase has released glucose and rhamnose, two of which occur naturally in the palmyra tuber. As a tetraglycoside of spirost-5en-3flol called flabelliferin11, the main bitter principle of palmyra fruit pulp is identified. Naringinase action, which results in a beverage with a pleasant mango cordial-like color, flavor, and texture, can remove bitterness. In various studies, the structural and biological properties of flabelliferin11 are reported [27-29].

The chemical constituents of Borassus flabellifer in-

clude gums, albuminoids, fats, steroidal glycosides, and carbohydrates like sucrose. The fresh pulp is reportedly rich in vitamins A and C. The fresh sap is reportedly a good source of vitamin B-complex. The male inflorescence constitutes borassosides and dioscin, spirostane-type steroid saponins [29]. Six new spirostane-type steroidal saponins, borassosides, were isolated from the methanolic extract together with 20 known steroidal glycosides ( $\beta$ -sitosterol 3-O-b-D-glucopyranoside), dioscin, to increase serum glucose levels in rats loaded with sucrose [11].

**Figure 4.** Structure of β-Sitsosterol and Flabelliferins [33]

Palmyra fruit pulp (PFP) extracted from the fruit is said to contain 0.42 g of amino acids per 100 g of pulp (dominated by lysine, phenylalanine, and glutamate). A rich source of carotenoids is the PFP. Although PFP is included in several recipes, due to the presence of a saponin known as flabelifferin11, use has been limited [30]. GC-MS was analyzed for the palmyra palm fruit bunch oil compositions. Campesterol, ethanol, 2- [2-(4 pyridyl) ethylamino], benzene-1,3,5-D3, phenol, and phenol derivatives were the major bio-oil compounds. As a result, pyrolysis products can be used as a source of hydrocarbons and can be used directly as a low-grade fuel or upgraded to high-quality liquid fuel. After separation, the components and their derivatives found in the GC-MS analysis are used for medicinal or chemical feedstock [31]. Alpha-glucosidase inhibitory activity was shown by palmyra palm extract and isolated compounds (tyrosol, and glucosyl-(6-1)-glycerol) [32].



Figure 5. Images of some important Palmyra components and representation of organic polymeric compounds in Palmyra palm

### 3. Conclusion

Palmyra culture is very helpful in achieving a self-reliant lifestyle and eco-friendly community life that leads to sustainable development. The Palmyra trees are unique in providing us with food and non-food products. They produce many useful polymers such as pectin, cellulose, hemicellulose, pentosan sulfate, polyphenol, and lignin. Although the palm tree is the official tree of Tamil Nadu, the government of Tamil Nadu has prohibited the production of palm toddy. Palm toddy has a high nutritional and medicinal value and is used as a therapeutic vehicle in the medicine of Tamil. The Tamil Nadu Government must therefore revisit the current policy and allow the people to produce, drink and sell palm toddy, as toddy is a natural drink, and therefore its extraction is a fundamental right under the Indian Constitution. Palmyra trees are destroyed as never before and have never been taken care of, and have always been taken for granted, despite various advantages and benefits. Efforts should be directed towards the planting of more trees, the protection of existing trees, the promotion of self-help groups for palmyra farmers (palmyra warriors), and the cultivation of more plants around water bodies. By producing more value-added products from Borassus flabellifer Linn, Linn provides an opportunity for a self-reliant lifestyle, eco-friendly community living, green economy, biodiversity/ecosystem, and sustainable development.

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