Chapter Four

Emulsions rheology of some *Acacia* gums and blends

4.1. General introduction to rheology

The rheological characteristics of a fluid are important in evaluating the ability of a fluid to perform a specific function. Many materials in use today are disperse systems where one substance (often particulate) is dispersed in another phase. These materials include adhesives, agrochemicals, cement, ceramics, colloids, cosmetics and personal care formulations, food and drink, mining and mineral slurries, paints, inks and surface coatings, pharmaceuticals and polymer systems. For example:

In the inks industry, the understanding of rheology and particle properties allows solid pigment content to be changed in different formulations whilst maintaining the critical rheological characteristics required for optimized printing. In the cement industry, the understanding of rheology and particle properties, such as the aggregate morphology, allows flow behavior during processing and application to be controlled. In the Cosmetics and Personal Care industries, it is essential to understand the relationship between rheology and particle properties to provide the optimum balance in terms of formulation, consumer acceptance and application performance. The physical properties of the dispersed particles, such as the average particle size, size distribution, the charge on the particles and even the shape of the

particles all help influence the overall, bulk, materials properties (Braun and Rosen, 2008; Satti, 2011).

Historically, the origins of Rheology can be traced back to a Pre-Socratic Greek metaphysician named Heraclitus. In the 5th Century B.C., Heraclitus was noted for documenting "Everything Flows."At the turn of the 17th century, renowned English mathematician and rheologist Sir Isaac Newton started to define rheology as a formal science. When a force is applied to a liquid, the liquid will flow to relieve the strain from this force. Different systems will resist this flow more than others and the measurement of this resistance is a measure of the viscosity of the system (Han, 2007; Satti, 2011).

4.1.1. Food emulsions rheology

Food emulsions exhibit a great diversity of rheological characteristics, ranging from low-viscosity Newtonian liquids to viscoelastic and plastic materials. Hydrocolloids are usually key components in food emulsions to deal with creaming instability. There is a growing emphasis on understanding the colloidal basis of the rheology of food emulsions. Viscoelastic measurements are appropriate tools for obtaining information about the microstructure of the system related to the organization of the macromolecules in the medium (Lorenzo, Zaritzky, and Califano, 2011). Hydrocolloids serve as emulsifiers and stabilizers in beverage emulsions. Hydrocolloids stabilize these emulsions through viscosity

effects, steric hindrance and electrostatic interactions. Hydrocolloids suitable for this purpose must have high solubility in cold water, low viscosity in solution, high emulsifying capacity and no thickening and/or gelling effects with aging (Buffo, Reineccius, and Oehlert, 2001). Gum arabic is the best-known hydrocolloid for use in beverage emulsions. Gum arabic is an unusual polysaccharide due to its excellent emulsifying properties and low viscosity, despite its high molecular mass, about 400,000 Da, (Buffo et al., 2001). The polyanionic character of gum arabic, coupled to the, highly, branched compact structure, confer a great cold solubility. Concomitantly, the viscosity of gum arabic solution is low compared to other polysaccharides of similar molecular mass, and flow behavior considered to be Newtonian up to 20-30% gum concentration or higher (Anderson and Rahman, 1967; Sanchez et al., 2002; Williams, Phillips, and Randall, 1990). The origin, nature and properties of the gum exudates of *Acacia senegal* var. *senegal* and *Acacia seyal* var. *seyal* have been described in previous papers. Both are acceptable within the regulatory specification of ''Gum arabic'' used in the food industry. (Al-Assaf et al., 2005; Al Assaf, Phillips, and Williams, 2005; Flindt, Al-Assaf, Phillips, and Williams, 2005; Siddig et al., 2005). *Acacia senegal* var. *senegal* is the main species used commercially due to its good emulsification properties for oil-in-water emulsions, cosmetic products, inks, etc. Generally, *Acacia seyal* var. *seyal* is less valued than *Acacia senegal* var. *senegal* due to its

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poor emulsification performance (Fauconnier et al., 2000; Flindt et al., 2005) and, therefore, tends to be used for applications where long term emulsion stability is not required(Elmanan et al., 2008).

4.1.2. Rheology definition

Rheology is the science of material deformation and flow. It is a branch of physics (and physical chemistry) since the most important variables come from the field of mechanics: forces, deflection and velocities. The term "rheology" originates from the Greek: "rheos" meaning "the river", "flowing", "streaming". Thus rheology is literally "flow science". However, rheological experiments do not merely reveal information about the flow behavior of liquids, but also the deformation behavior of solids. The connection here is that large deformations produced by shear forces cause many materials to flow(Mezger, 2006). It relates to the relationship between stress, strain and time. It would, commonly, use the words lubricate, spread, squeeze, as non-technical examples of rheological responses. As force is applied to an object it can do one of several things: It can cause the object to be strained or ruptured, or it can make the object move, flow or accelerate. Rheology describes material response to the forces of deformation. (Goodwin and Hughes, 2008). Newton first introduced a basic model for the flow measurement of a liquid between two parallel plates and this is represented in Figure (4.1). Using this model a number of common rheology terms can be defined as follows:

4.1.2.1. Shear Stress (τ)

Shear stress (τ), is the force required to move a given area of the fluid,

$$\tau = F/A \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (4.1)$$

With the (shear) force F [N] and the (shear) area A [m^2].

Where 1 N (Newton) =1kg.ms^{-2}

The unit of the shear stress is Pascal [Pa] or [Nm^{-2}].

4.1.2.2. Shear Rate (γ)

Shear Rate (γ), is the rate of movement of the fluid between the plates. It is determined by dividing the velocity difference between the plates by the distance between them. This defines the velocity gradient,

$$\gamma = dv/dx \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (4.2)$$

With the velocity v [m/s] and the gap between the plates x [m].

The unit of the shear rate is [1/s] or [s^{-1}].

4.1.2.3. Viscosity (η)

It is the measurement of the resistance to flow of a fluid and is expressed mathematically as,

$$\eta = \tau/\gamma \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (4.3)$$

Where viscosity (η) = shear stress (τ)/shear rate (γ).

Pascal second (Pa.s) is the basic unit of viscosity, but Poise or centipoises (cp = one hundredth of a Poise) is often used and one cp is equivalent to a millipascal-

second, mPa.s. When quoting viscosities the shear rate (or measurement method/equipment used) should be stated together with the temperature at which the measurement was taken. Rheological measurements are generally represented by a plot of log η versus log γ, logarithm is taken to enable the viscosity shear rate data to be presented over many orders of magnitudes (Goodwin and Hughes, 2008).

4.1.3. Rheological properties

The rheological properties of a liquid are dominant features that can be quantified to characterize its behavior, and the response of a liquid to a forced shearing flow is the basis for determining the specific rheological properties of a given liquid. General qualitative terms used to describe these properties are viscoelastic, Newtonian, non-Newtonian, thixotropic, and dilatants. Quantitative parameters used are viscosity, elasticity, shear rate, shear strain, and shear stress. The broadest view of liquid rheology is obtained by using oscillatory flow at a selected frequency because both viscous and elastic properties are revealed. Steady flow reveals only viscous properties. Values of shear stress, shear rate, and shear strain are primary parameters for quantitative specification of both the flow condition and the liquid response. It is from these quantities that the components of the viscoelastic modulus, the elasticity and the viscosity (or alternatively the storage

and loss moduli) are obtained. These numbers form the basis for quantitative specification of the liquid's properties for quality control or other applications.

4.1.4.   Rheological Classifications of emulsions

All forms of shear behavior, which can be described rheologically in a scientific way, can be viewed as lying in between two extremes: the flow of ideal viscous liquids on one hand and the deformation of ideal elastic solids on the other. Two illustrative examples for the extremes of ideal behavior are a low viscous mineral oil and a steel ball. There are two types of fluids: Newtonian and Non- Newtonian.

4.1.4.1.   Newtonian ideal liquid emulsions

Newtonian ideal liquid emulsions have true viscous flow, which means the shear stress is, directly, proportional to shear rate and the viscosity is independent of the shear rate within the laminar flow range and the plot begins at the origin. Examples of the Newtonian emulsions are tea, coffee, sugar syrups, beer, carbonated beverages, filtered fruit juices, edible oils, and milk. Dynamic viscosity and coefficient of viscosity are synonyms of the term 'viscosity' when referring to Newtonian emulsions. They exhibit simplest types of flow properties. All other types of food are non-Newtonian. Here

$$\tau = \eta\gamma \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4.4)$$

Where; τ is the shear stress, η is the viscosity and γ is the shear rate.

4.1.4.2.   Non-Newtonian emulsions

Many systems show behavior that deviates from that of simple Newtonian flow. The viscosity of the emulsions depends upon applied shear rate (time independent) and/or time over which shear stress is applied (time dependent):

4.1.4.2.1.   Time independent non-Newtonian Fluids

i. Plastic (Bingham) fluid emulsions: Such a behavior is displayed by fluids which behave like a solid until a finite shear stress known as yield stress (τ_0) is applied to begin the flow. Elastic deformation is exhibited at stress below the yield value while the plastic flow is observed at high shear stress. Some examples of this kind of fluid emulsion are mayonnaise, whipped cream, and margarine.

ii. Pseudo-plastic or shear thinning fluids: Here the apparent viscosity of the emulsion decreases as the shear rate is increased. The curve begins from the origin but concaves upward. Shear-thinning may be thought of being due to breakdown of structural units in a food due to the hydrodynamic forces generated during shear. Most non-Newtonian food emulsions exhibit shear-thinning behavior, including many salad dressings and some concentrated beverages (Rao, 1999).

iii. Dilatant or shear thickening fluid emulsions: The shear stress-shear rate plot of this type of a flow begins at the origin but equal increments in the shear stress gives less than proportional increase in the shear rate. There is an apparent increase in viscosity with increasing shear rate, e.g. high solids, raw starch suspensions,

chocolate syrups, although dilatant flow is rarely seen in food industry and finished food products.

In all the above three cases, viscosity at any given shear rate can be calculated by

$$\eta = \tau_0 + m\gamma^{n-1} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (4.5)$$

All the above flow behaviors can be represented by this equation as represented in Figure (4.2). Newtonian flow is represented by a straight line starting from the origin, dilatant flow starts from the origin and concaves downward whereas Pseudoplastic flow starts at the origin and concaves upward. Plastic or Bingham flow is linear and does not begin from the origin, has some yield stress before the liquid flows. For Herschel-Bulkley, the flow behavior above the yield value of a plastic fluid is not linear.

4.1.4.2.2. Time dependent fluid emulsions

Time dependent fluid emulsions exhibit shear rate as a function of both magnitude and duration of shear, and time lapse between two successive shearing actions (Figure 4.3).

i. Thixotropic flow: The apparent viscosity decreases with shearing time but the change is reversible. This phenomenon is explained by the breakdown of forces that exist between particles of a system which return back to original state on standing, e.g. starch gel pastes, starch-thickened baby food or yogurt.

ii. Rheopectic flow: The apparent viscosity increases with time of shearing and the change is irreversible, e.g. egg white foam and whipped cream. When egg white is beaten, its viscosity increases until it becomes stiff, in other words it thickens.

4.1.4.3. Viscoelastic emulsions

Emulsions which exhibit both, viscous and elastic properties simultaneously are viscoelastic (Bourne, 2002). When a deforming force is applied on a viscoelastic emulsion it continues to deform as long as the force is pressing against it. When the force is removed there is some recovery of the original shape, due to elastic component, also calculated as storage G' (or elastic) modulus. But the recovery is not complete because of the viscous component. Due to this partial recovery, some energy is lost which is defined as the loss G'' (or viscous) modulus. Rheological properties of viscoelastic materials are therefore characterized as

$$G^* = G' + iG'' \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \quad (4.6)$$

Where; G' is known as the storage modulus and G'' as the loss modulus (Arora, 2011).
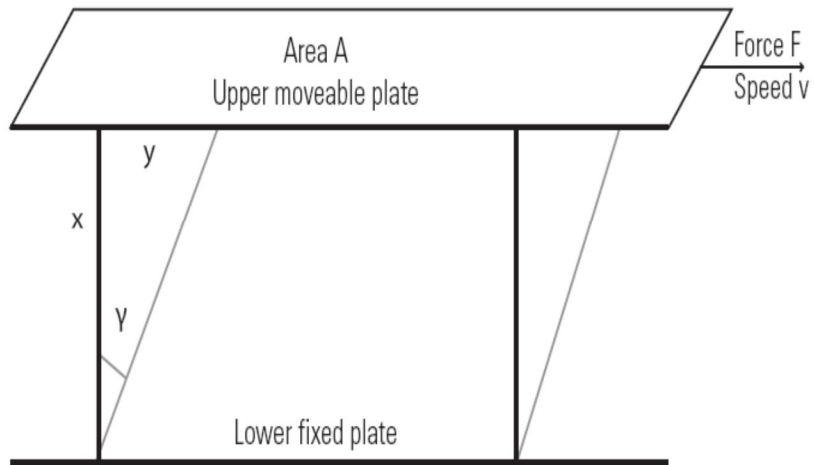
Figure (4.1): Diagram for rheological definitions.
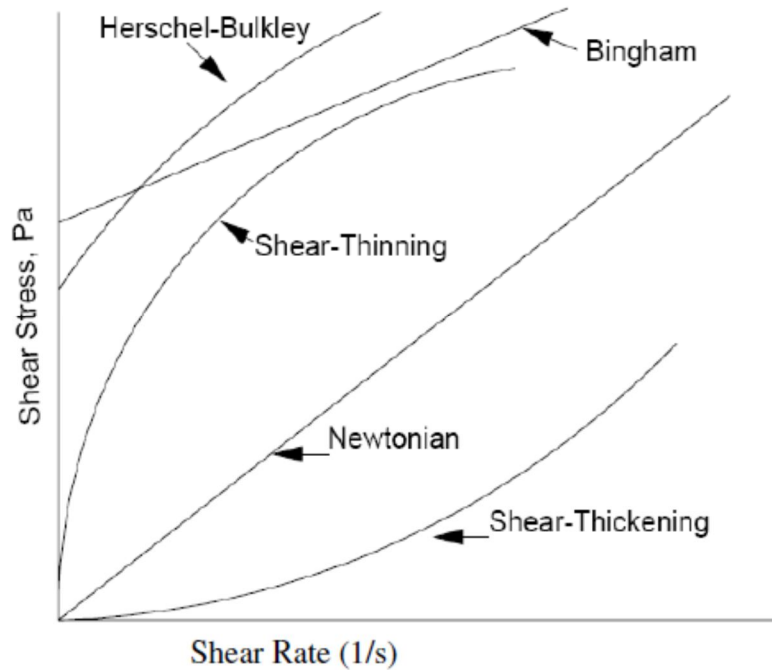
Figure (4.2): Basic shear diagram of shear rate versus shear stress for the classification of time independent flow behavior of pseudoplastic and dilatants fluid (Bourne, 2002).
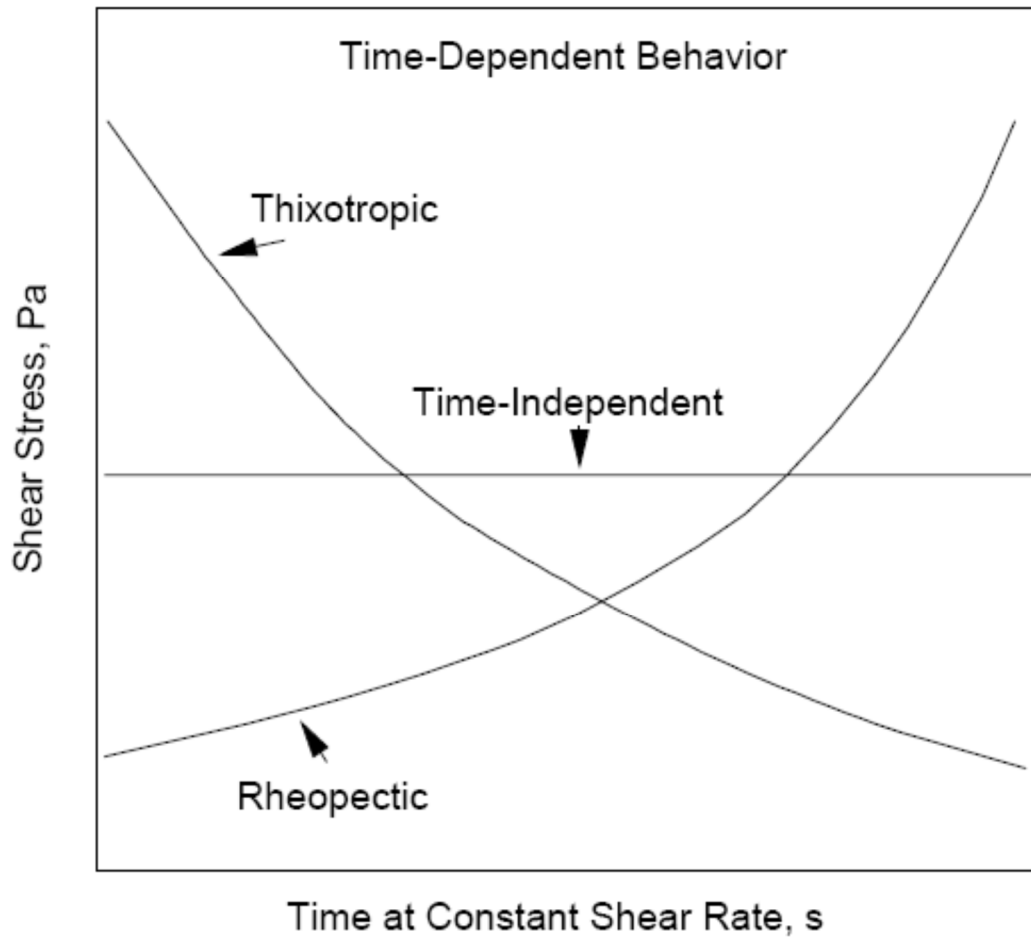
Figure (4.3): Time dependent non-Newtonian fluids (Bourne, 2002).

4.1.5.   Rheological behavior of emulsion

The rheological behavior of emulsions has been of great interest not only for fundamental scientific understanding but also for practical industrial applications (Barnes, 1994; Tadros, 1994). However, emulsions exhibit contrasting rheological behavior to that of suspensions of hard spheres, due to the droplet deformation, the surface mobility and the internal fluid circulation. Since the rheological response of bulk matter to applied mechanical force is rooted in the interaction of its constituent molecules, the science of rheology makes a useful tool in this study. The same consideration, of course, applies to the rheological behavior of colloidal dispersion especially in cosmetic (emulsion). Commonly, these rheological or flow studies were contributed by two major measurements with steady state rotational shear (Viscometry) and (Oscillatory) or small amplitude oscillation. (Barnes, Hutton, and Walters, 1989; Walters, 1975).

Rheology of beverage emulsions is a subject of high importance for industrial applications. Primarily, the rheology of emulsions is a direct manifestation of the various interactions that occur within an emulsion system (Tadros, 1994). Understanding the rheological behavior of emulsions has been of great interest for its strong relationship to many properties of emulsions that are vital for various applications in food industry. Creaming of oil droplets in beverage emulsions is strongly dependent on the viscosity of the component phases.

4.1.6.   Factors affecting emulsion rheology

Factors affecting emulsion rheology can be summarized as:

1. Dispersed phase volume fraction: One of the most important factors which affect emulsions rheology is the oil phase volume fraction. The viscosity of an emulsion increases, linearly, with oil phase volume fraction.

2. Viscosity of the dispersed droplets: Another factor that may affect the rheology of the emulsions is the viscosity of the dispersed droplets. The viscosity of an emulsion increases linearly with dispersed phase volume fraction.

3. Droplet size distribution: The third factor that affects emulsion rheology is the droplet size distribution and polydispersity. The effect of both droplet size and droplet size distribution on the rheology of an emulsion depends upon oil phase volume fraction and the nature of the colloidal interactions. At a critical distance of separation between the droplets, which depends on droplet size, the viscosity shows a rapid increase.

4. Continuous phase: Three main properties of the continuous phase must be considered. The first and the most important is the viscosity of the medium. The viscosity of the emulsion is, directly, proportional to the viscosity of the water phase. Addition of additives such as excess emulsifiers and thickeners (e.g. polysaccharides) may also affect the viscosity. The second property of the medium is the chemical composition such as polarity and pH which affect the charge on the

droplets and hence their repulsion. The viscosity of the emulsion is, directly, related to the magnitude of the repulsive forces. The latter is also affected by the nature and concentration of electrolyte in the system, which represents the third important attribute of the aqueous phase.

5. Interfacial film: The rheology of emulsion may also be influenced by the interfacial rheology of the emulsifier film surrounding the droplets. When shearing is applied to an interfacial film, its constituent molecules as well as the molecules of the oil and water phases in its immediate vicinity are displaced from their equilibrium positions. This will have an effect on the interfacial viscosity of the film, consequently on the bulk rheology of the emulsion, if the latter is formed from large deformable droplets. The nature and concentration of the emulsifier, in particular, its solubility and distribution in both phases, also have a large effect on the rheology of the system (Friberg, 2004; McClements, 2004; Tadros, 1994). Hence, rheological characterization of emulsions provides important information to improve and optimize production and manufacturing processes of emulsions. Parameters from rheological tests are interwoven, complement each other, and describe properties of formulations during processing, application and storage of emulsions. Therefore, reliable research and a thorough understanding of rheological properties of emulsions are necessary (Arora, 2011).

The objectives of this work are

- To measure the rheological properties of some *Acacia* gums (ASG, ASY, ATR, and AMF).

- To measure emulsions rheological properties of the above *Acacia* gums.

- To measure emulsions rheological properties of the blends of gums.

4.2. Materials and methods

4.2.1. Materials

Description of materials was given in chapter two and three of this work.

4.2.2. Preparation of gum solution

Deionized water was added to an appropriate weight of gum sample (based on dry weight) to give 50-20% (w/w) gum concentration in 100Ml glass bottle, sample was dissolved using magnetic stirrer for 3hrs and left overnight for full hydration then, centrifuged using (VELOCITY 18R REFRIGERATED CENTRIFUGE, Dynamica) for10 min. at 2500 rpm to remove insoluble particles and air bubbles(Sanchez et al., 2002). The solution was filtered using 100 μm mesh and stored at 4**C**.

4.2.3. Preparation of gum's emulsion

Varying amounts of IPM oil were added to an appropriate amount of 30% aqueous gum solution to make 20%-10% (w/w) oil concentration and (20% w/w) gum concentration. Emulsions were prepared using (ULTRA TURRAX T25 basic IKA)

homogenizer at 24,000 rpm for 5min. Emulsions formed were kept in incubator of 45.0±0.1**C** for one day before analysis.

4.2.4.   Preparation of gum blend's emulsions

All gum blends emulsions were prepared with an optimum IPM oil concentration (20%) and 20% total gum content.

The *Vulgares* gums (ASG and AMF) were blended with 1:1 formula and a description of the preparation formula for ASY and ATR from the *Gummiferae* series was given in Table (3.1).

4.2.5.   Flow and dynamic rheological measurements

For rheological measurements CVO-R Rheometer Germini^TM, Malvern Instrument UK with temperature regulator of Peltier Plate system (-40°C to 180°C, from Bohlin Instrument Ltd) was used. The sample was placed in 40mm cone geometry of 4° and solvent trap at 25**C**. Rheological measurements were measured for the four *Acacia* gum solutions at different concentrations, emulsions at varying levels of IPM oil concentration, and emulsions of gums blends with different proportions. Measurements were done after one day of incubation at 45**C** of the emulsions. Flow curves were determined at increasing shear rate (0.01-1000 s^{-1}) within 8 minutes. Viscosity was calculated as a function of shear rate. For dynamic or oscillatory measurements, a frequency sweep from 0.1 up to 10 Hz was given at fixed oscillation strain and G′ (elastic modulus) and G″ (viscous modulus) were

obtained. Dynamic measurements were conducted to describe the rheological properties of emulsions more clearly. The storage modulus (G′), loss modulus (G″) and phase angle (δ) are among the parameters that characterize a system in a dynamic rheological study (Al-Assaf, Phillips, Aoki, and Sasaki, 2007). The region of linear viscoelasticity (LVR) was determined prior to each frequency sweep performing amplitude (strain) sweeps to verify the linear relationship between stress and strain. G′ is a measure of the energy stored in a cycle of oscillation whereas G″ is the measure of energy lost as viscous flow in a cycle of oscillation. The focus was to study the storage modulus (G′) and loss modulus (G″). For a, pure, viscous system, G′ = 0 and G″ = G*, where G* is the complex modulus. Conversely, G′ = G* and G″ = 0, if the system is purely elastic.

4.3. Results and discussion

4.3.1. Rheology

The rheological properties of *Acacia* gums solutions, emulsions and blend's emulsions where investigated.

4.3.2. Flow rheological measurements

The flow curves of gums solutions (Figures 4.4-4.6), showed a, nearly, Newtonian flow for the 50% and 40% ASG and 50% ASY gum concentrations. But for the lower concentrations the flow curves, at increasing shear rate, showed that the viscosity was decreasing; a plateau was reached for shear rate above $10s^{-1}$. Similar

shear-thinning flow behavior has been reported (Mothe' and Rao, 1999; Sanchez et al., 2002; Williams et al., 1990). Flow curves of the gums solutions at increasing or decreasing shear rates were similar above $1\text{-}10\text{s}^{-1}$, below these shear rates flow curves at decreasing shear rates were, slightly, above flow curves at increasing shear rates, indicating higher viscosities of solutions. This effect was reproducible and could be explained by the development of a shear-induced structure. When decreasing the shear rate to low values, the association equilibrium would shift to aggregation again, during which new architecture of the aggregates are rebuilt, inducing the viscosity of shear rate ramp- down to be higher than ramp-up stage. Figure (4.7) describes this change in architecture of the aggregates during shear rate ramp-up and ramp-down(Li et al., 2009).
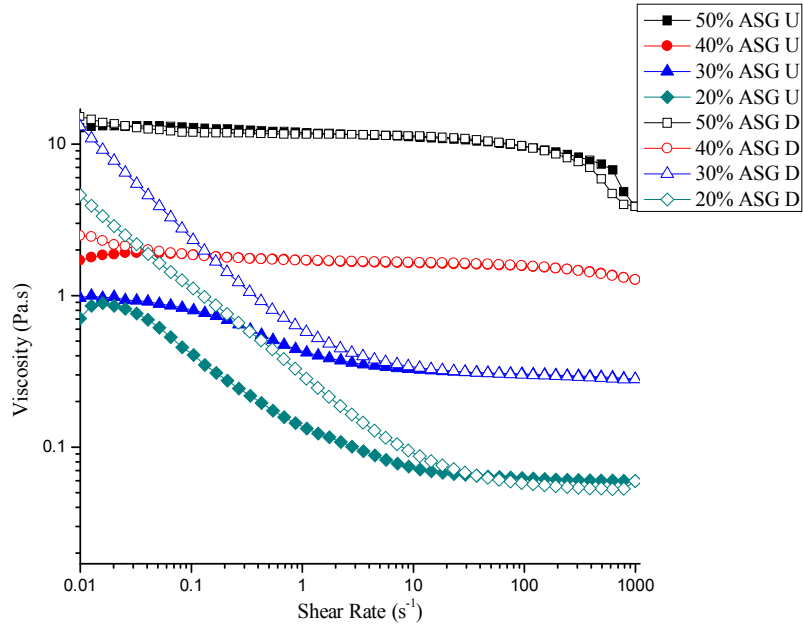
Figure (4.4): Flow curve at increasing (U) and decreasing (D) shear rate obtained on 50%, 40%, 30%, and 20% ASG gum solutions.
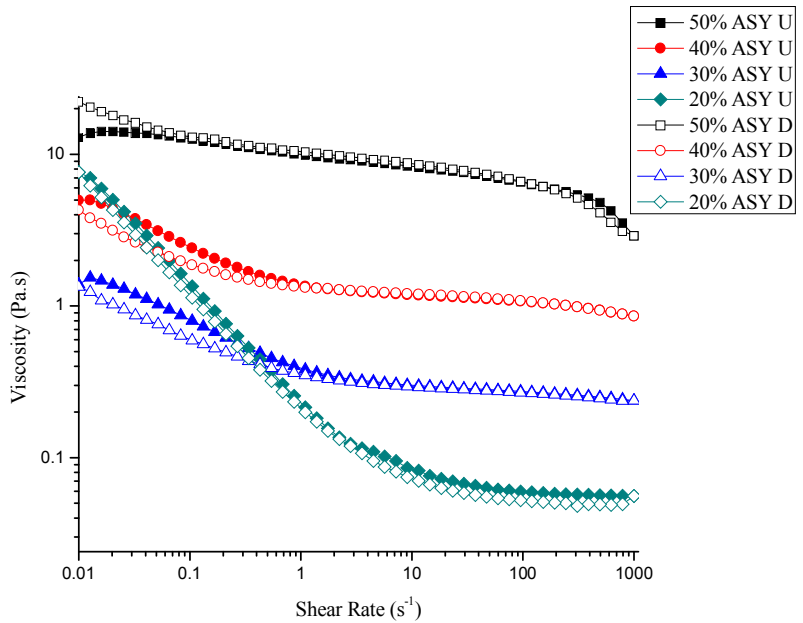
Figure (4.5): Flow curve at increasing (U) and decreasing (D) shear rate obtained on 50%, 40%, 30%, and 20% ASY gum solutions.
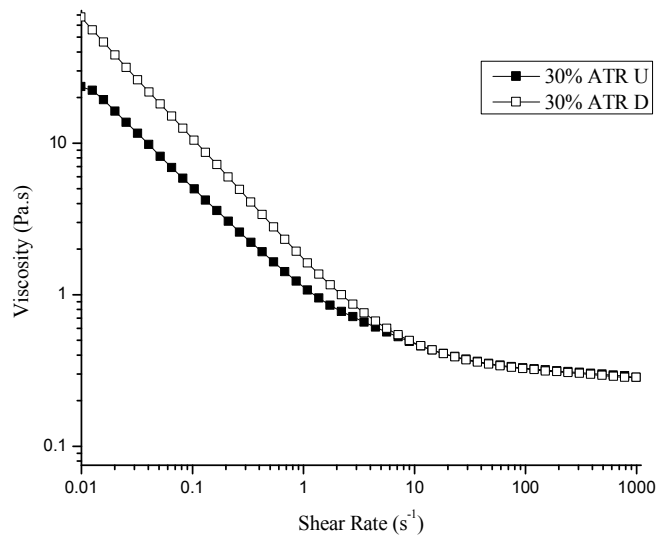
Figure (4.6): Flow curve at increasing (U) and decreasing (D) shear rate obtained on 30% ATR gum solution.
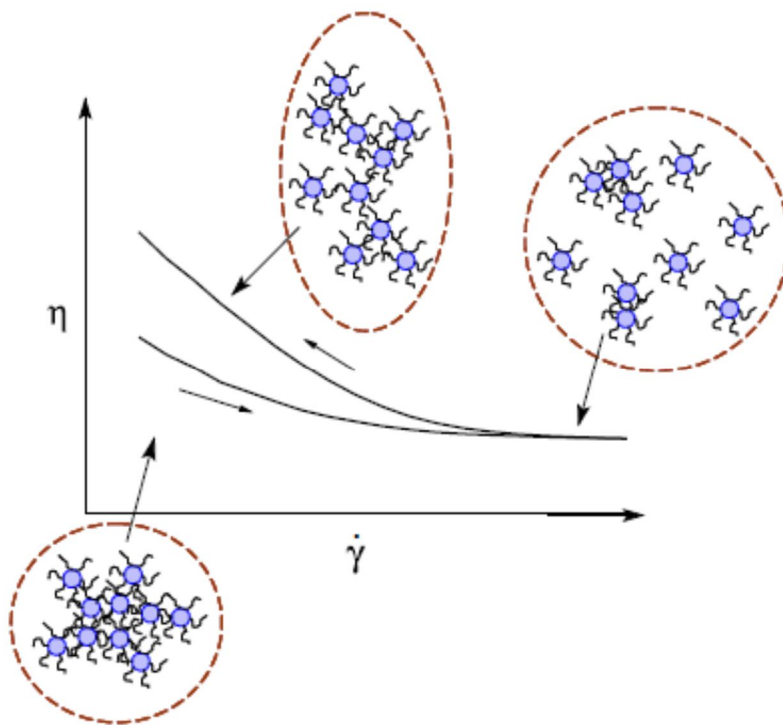

Figure (4.7): Architectural change of the aggregates during shear upon increasing shear rate and decreasing shear rate (Li et al., 2009).

Figures (4.8-4.11) showed the flow curves of ASG, AMF, ASY, and ATR gums emulsions. The four species of *Acacia* gum emulsions showed decrease in viscosity with the decrease of oil concentrations from 20% to 10%, AMF gum emulsions showed higher viscosities than the other gums. AMF and ASY emulsions behaviors were, nearly, Newtonian and the viscosity remains constant at entire shear rate range for the high oil concentrations. Gums emulsions show shear-thinning behavior at low shear rate and a Newtonian behavior at high shear rates. This behavior is affected by the molecular association exhibited at low shear rate where viscosity is dominated by the elastic contribution reflecting the existence of molecular association in the continuous phase, while at higher shear rate above $50s^{-1}$ only the viscous contribution dominated with complete breakdown of molecular association. At this condition *Acacia* gums emulsions exhibited Newtonian behavior, these results agrees with the findings of Jayme, Dunstan, and Gee (1999) who affirm that *Acacia* gums impart stability to emulsions through an electrosteric mechanism with the dominance of the steric contribution.

This shear-thinning or, alternatively, pseudoplastic behavior of emulsions was due to gums flow behavior, as similar behaviors were observed from gum solutions (20% and 30%). Gum arabic like other high-molecular-weight polymers such as beta-lacto globulin and xanthan showed pseudoplastic behavior (Mandala, Savvas, and Kostaropoulos, 2004). The systems contain highly entangled long chain

polymeric molecules in solutions like gum arabic, are oriented randomly corresponding to their minimum energy state. At low levels of, externally, applied stress, the system resists any deformation and strives to retain its structure, thereby offering a very high resistance by exhibiting a very large value of the apparent viscosity (Mandala and Bayas, 2004). As the level of shearing stress is gradually increased, the structural units respond to the applied stress either by aligning themselves in the direction of flow, or by deforming to orient along the streamlines, or by way of decomposition of the aggregates into, primary, particles. Similarly, coiled and entangled polymeric molecules may become disentangled and finally, may fully straighten out. All these micro-structural changes facilitate bulk flow in the system. This can be seen as the lowering of the, effective, viscosity with increasing shear rate, as seen in viscoelastic and shear-thinning substances (Mandala and Bayas, 2004; Radi and Amiri, 2013).
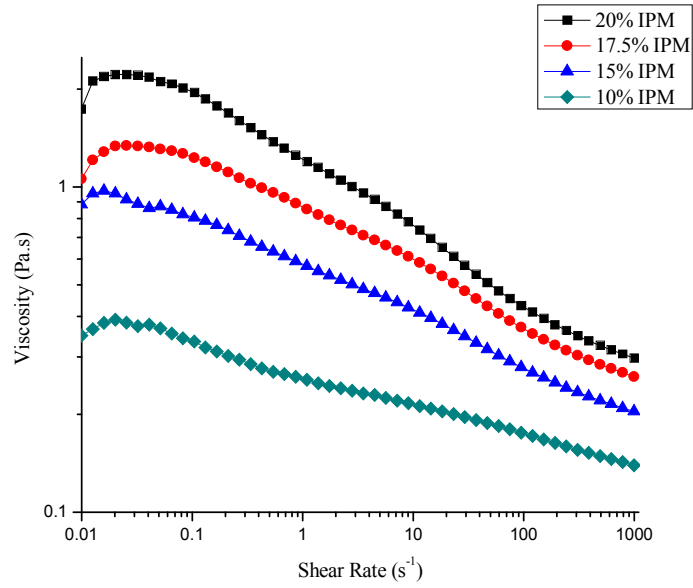
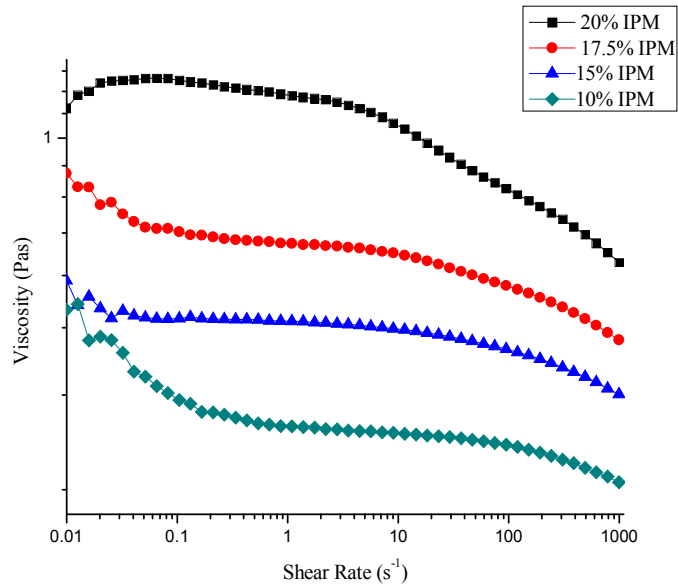**Figure (4.8): Viscosity profile of ASG gum emulsion with different IPM oil concentrations (20% -10%).**

Figure (4.9): Viscosity profile of AMF gum emulsion with different IPM oil concentrations (20%-10%).
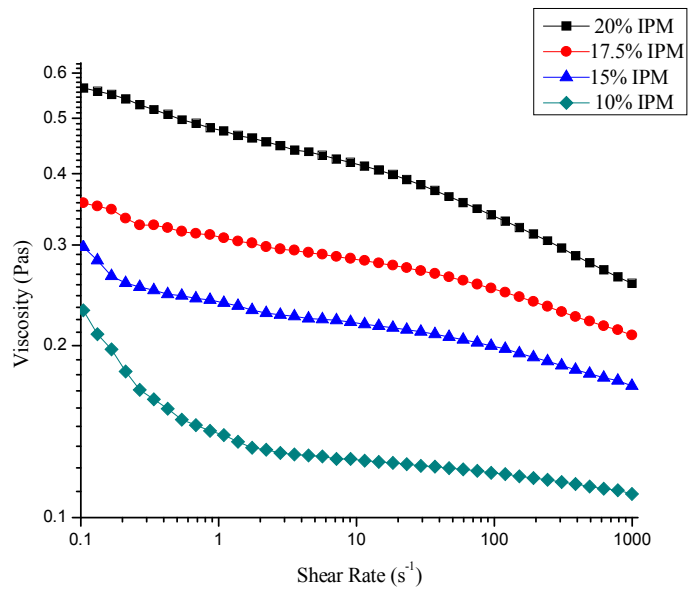
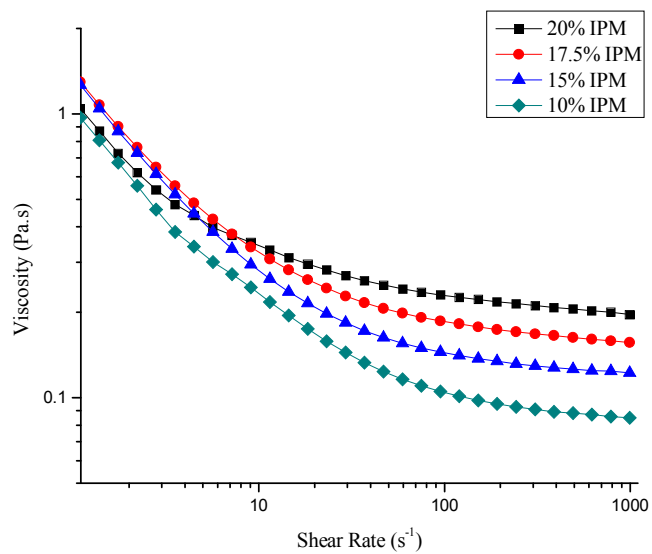**Figure (4.10): Viscosity profile of ASY gum emulsion with different IPM oil concentrations (20%-10%).**

Figure (4.11): Viscosity profile of ATR gum emulsion with different IPM oil concentrations (20%-10%).

Figures (4.12 and 4.13) reflected the flow behavior of gums blends emulsions. It is found that the emulsion formed with the 1:1 ASG: AMF gums blend exhibited an intermediate flow behavior (shear-thinning) between the two gums emulsions (Figure 4.12). For ASY and ATR gums blends emulsions, the flow behaviors were varying from Newtonian to shear-thinning. And it was observed that the more ATR gum proportion the more pronounced is the shear-thinning behavior. The 3:7 ASY: ATR gum formula exhibited the higher viscosity reflecting good stability (Figure 4.13).
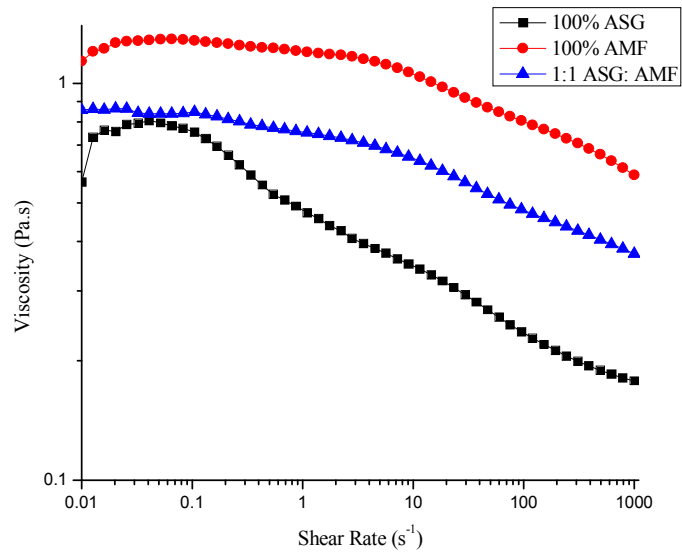
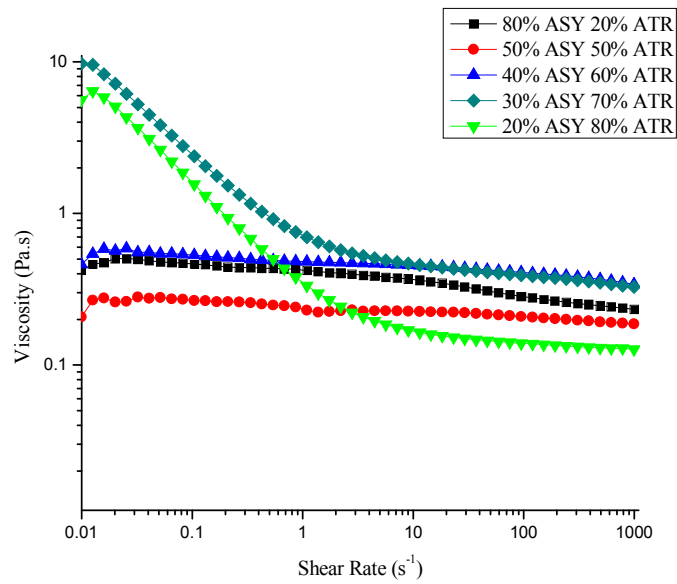**Figure (4.12): Viscosity profile of emulsions formed by ASG, AMF, and 1:1 gums blend at 20% IPM oil concentration.**

Figure (4.13): Viscosity profile of emulsions formed by ASY and ATR gums blends at 20% IPM oil concentration.

4.3.3.   Dynamic rheological measurements

Figures (4.14-4.16) represent the dynamic behavior of ASG and ASY gums solutions at concentrations varying from 50%-20%, and 30% ATR gum solution. The results indicate that with increase gums` solutions concentration, there was an increase in the viscoelastic properties. In all the cases G″, the viscous modulus, was found to be more dominant than G′, the elastic modulus, indicating the predominance of viscous liquid like behavior over solid elastic like behavior and that both moduli were frequency dependant (Al-Assaf et al., 2007). However the elastic modulus, sharply, drops before rapidly increases again. This point shifts to a higher frequency and higher storage modulus value as the gum concentration increases but was not observed for the 50% gum concentration. This behavior may be attributed to onset of disentanglement of polymer molecules of the gum at each, particular, point and regain of structure forming a network. These results are in good agreement with previous rheological flow measurements.

The effect of the gums under study on emulsions was studied using oscillatory measurements (G′ and G″ vs. frequency) within the linear viscoelastic range (LVR) previously determined by oscillation amplitude-sweeps. Figure (4.17) show the amplitude-sweep for ASG gum emulsions at different IPM oil concentrations.
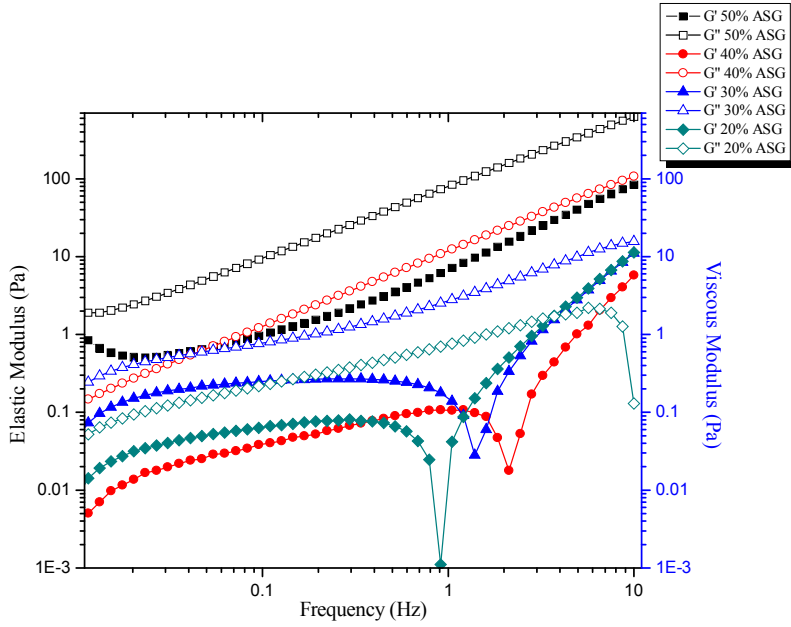
Figure (4.14): Oscillation frequency sweep test of 50%, 40%, 30%, and 20% ASG gum solution.
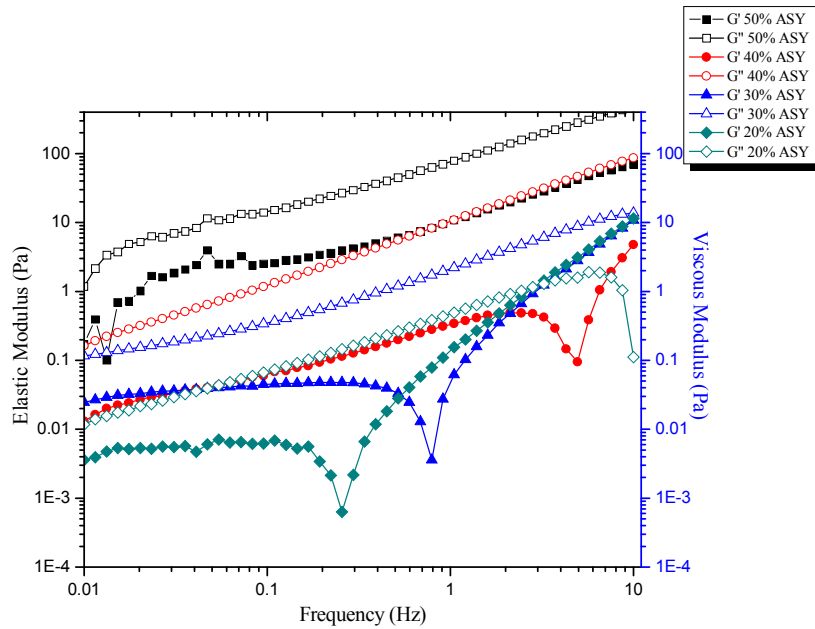

Figure (4.15): Oscillation frequency sweep test of 50%, 40%, 30%, and 20% ASY gum solution.
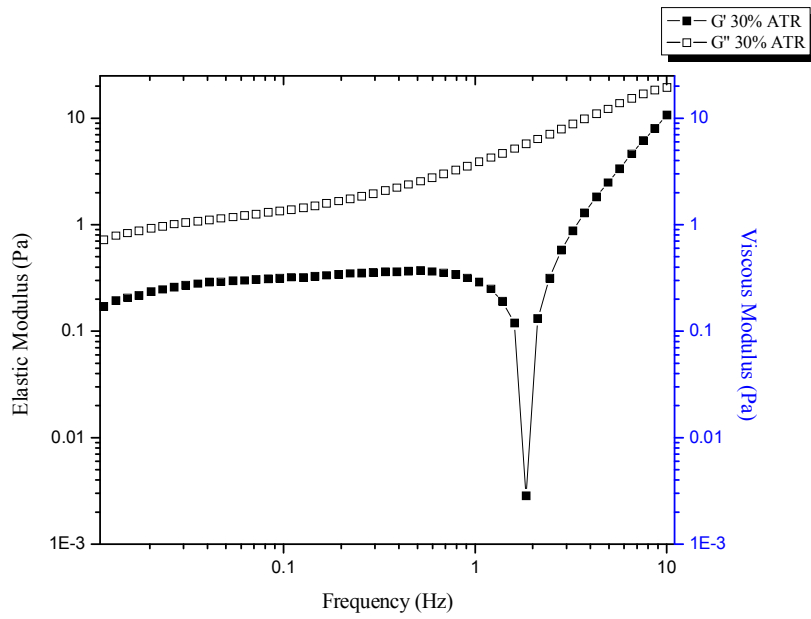
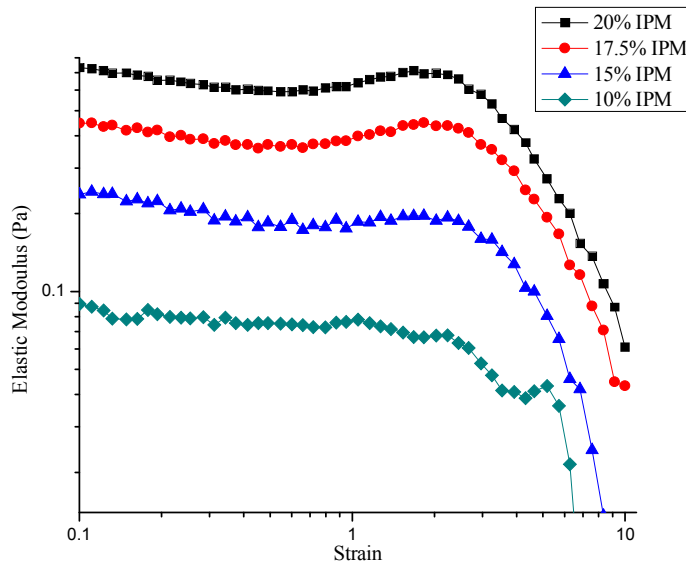**Figure (4.16): Oscillation frequency sweep test of 30% ATR gum solution.**

Figure (4.17): Oscillation amplitude-sweep for ASG gum emulsions at different IPM oil concentrations.

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Emulsions' oscillation frequency sweep at the studied IPM oil concentrations shows elastic and viscous moduli frequency dependence. However the elastic modulus, sharply, drops before rapidly increases to values approaching that of viscous modulus at 10 Hz. This point shifts to a higher frequency and higher storage modulus value as the oil concentration increases. This behavior may be attributed to onset of disentanglement of polymer molecules of the gum at each particular point and regain of structure forming a network around the oil droplets which do not themselves ruptured as the test was performed at the linear viscoelastic region of the emulsions. At this point droplet might also be elongated but no rupture or deformation is to be expected. For all gums emulsions the viscous modulus dominants over the elastic modulus which implies a viscous like behavior (Valdez et al., 2006). The ATR gum emulsion showed the highest moduli values followed by AMF, ASG, and ASY gum emulsions as in Figures (4.18-4.21).
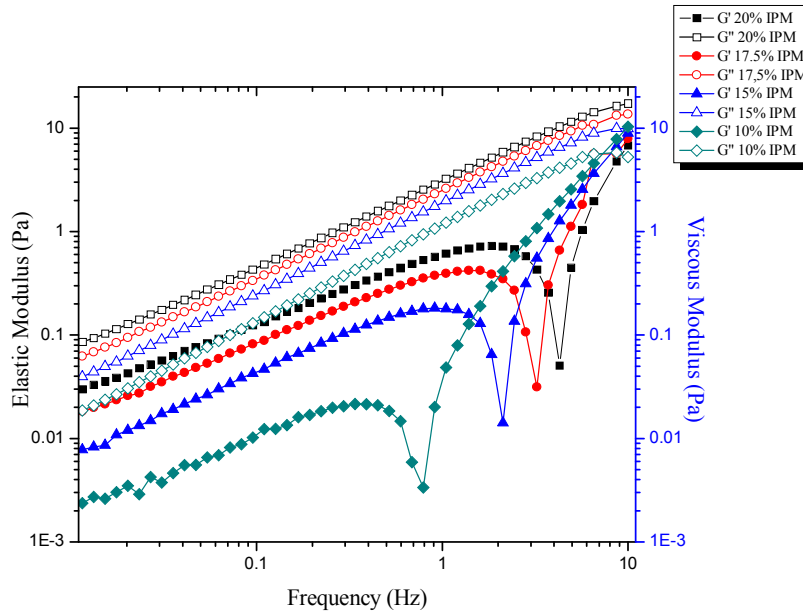
Figure (4.18): Oscillation frequency sweep test of ASG gum emulsion with 20%-10% IPM oil concentrations.
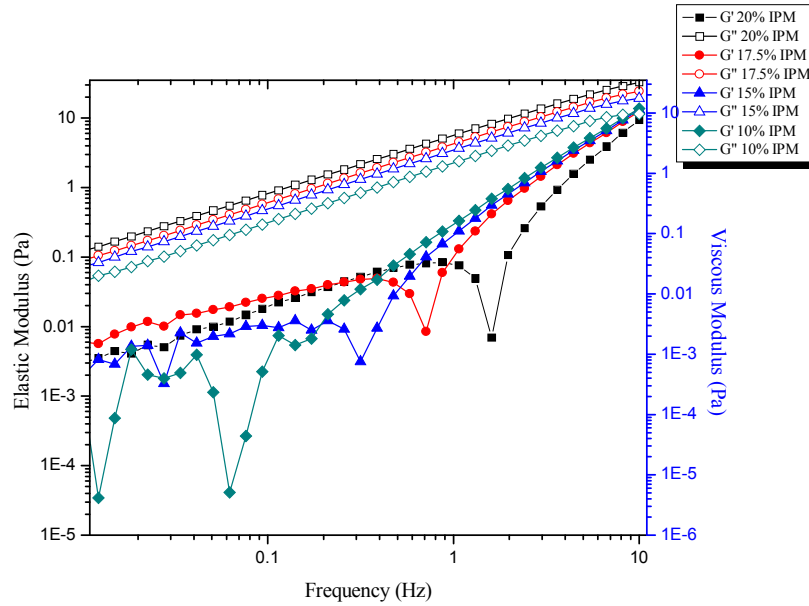

Figure (4.19): Oscillation frequency sweep test of AMF gum emulsion with 20%-10% IPM oil concentrations.
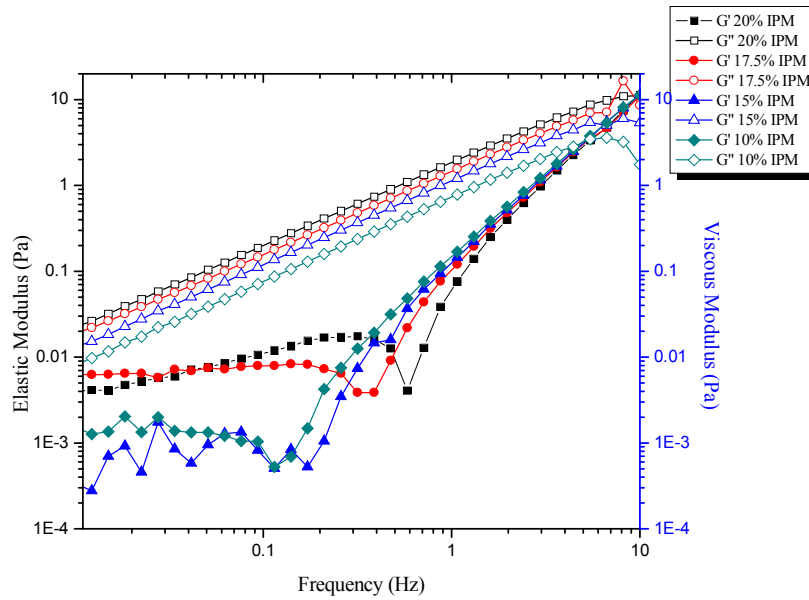
Figure (4.20): Oscillation frequency sweep test of ASY gum emulsion with 20%-10% IPM oil concentrations.
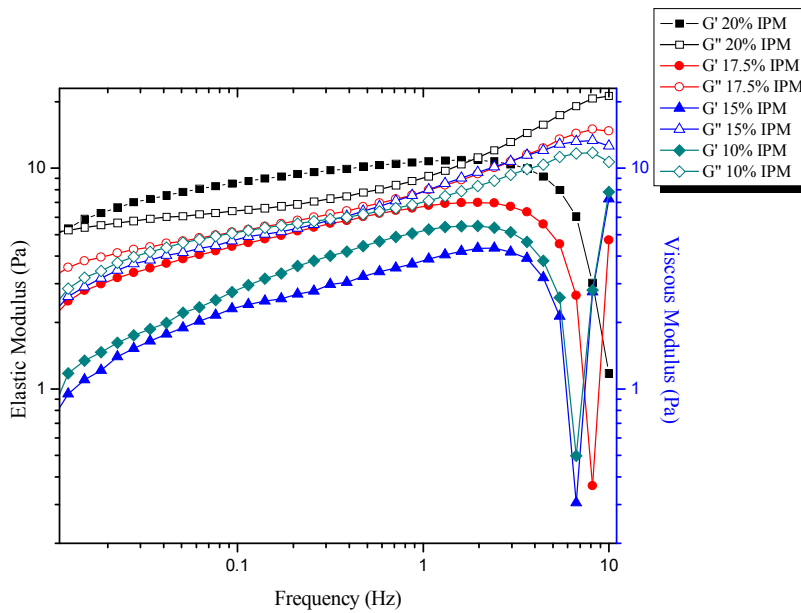
Figure (4.21): Oscillation frequency sweep test of ATR gum emulsion with 20%-10% IPM oil concentrations.

Figures (4.22 and 4.23) show dynamic rheological measurements of the emulsions formed by ASG and AMF 1:1 gums blend and ASY and ATR gums blends at varying proportions. The blends' emulsions behaviors were similar to that of gums solutions and emulsions. It is found that the 1:1 ASG: AMF gums blend emulsion showed higher viscoelastic properties than that of the individual gums emulsions. As for the ASY and ATR gums blends the best proportion with the highest viscoelastic properties was found to be the 3:7 formula.

Figure (4.22): Oscillation frequency sweep test of emulsions formed by ASG, AMF, and 1:1 gums blend with 20% IPM oil concentration.

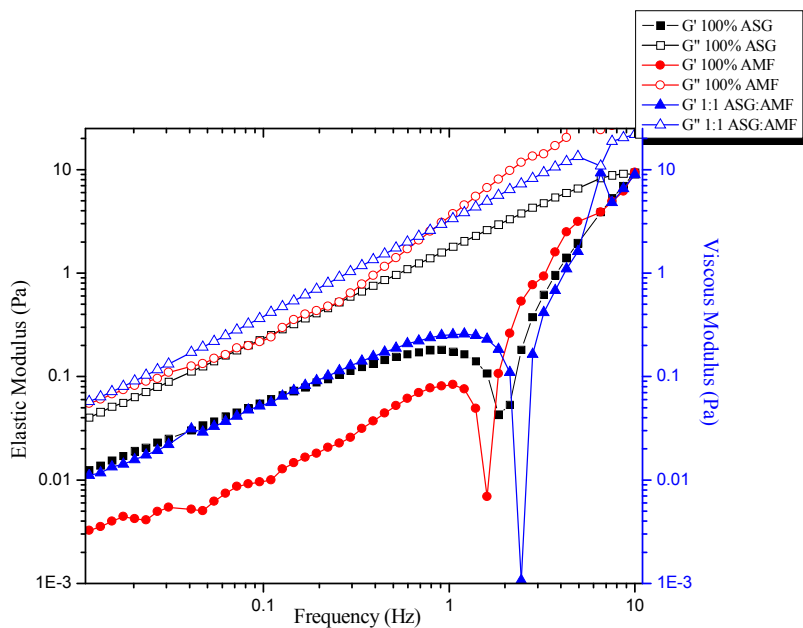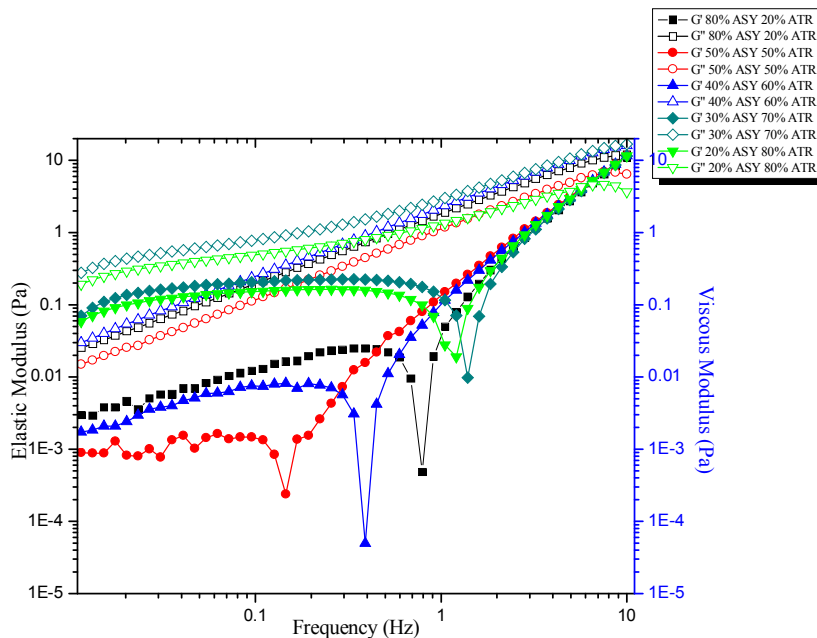**Figure (4.23): Oscillation frequency sweep test of emulsions formed by ASY and ATR gums blends with 20% IPM oil concentration.**

4.4. Conclusions

- According to these investigations the viscosity, oscillatory and rheological measurements provide a robust methods of emulsions characterization.

- The rheological characteristics of the investigated emulsions showed a dependency on the type of the used gum, as well as on the volume of the dispersive phase.

- Viscosity increased with the increasing volume of the dispersed phase.

- The emulsions exhibited Newtonian or pseudoplastic behavior at lower oil concentrations and nearly Newtonian at higher concentrations.

- The gums blends emulsions showed enhanced flow and dynamic rheological behaviors.

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