MAPAN-JOURNAL ,
Publisher: METROLOGY SOC INDIA , NPL PREMISES,
Address: DR K S KRISHNAN MARG, NEW DELHI, INDIA,
ISSN / eISSN:0970-3950 / 0974-9853
Volume 25-Issue 1-(2025)
https://doi-001.org/1025/17613073918924

OPEN ACCESS

Non-Destructive Testing and Rheological Properties of Soft Dielectric Materials

Kaddour Benyahia [1], Arsalane Chouaib Guidoum [2], Abdelhamid Benhaya [3]

1 Energy and Materials Laboratory, University of Tamanghasset, 11000, Tamanghasset, Algeria.
Email: kaddour.benyahia@univ-tam.dz

[2]Energy and Materials Laboratory, University of Tamanghasset, 11000, Tamanghasset, Algeria.
Email: acguidoum@unvi-tam.dz
[3]LEA, Department of Electronics, University of Batna 2, 05000, Batna, Algeria.Email:
a.benhaya@univ-batna2.dz

*Corresponding author(s). kaddour.benyahia@univ-tam.dz

Received: 01/06/2024 ; Accepted:10/10/2024 ; Published:28/12/2024

Abstract

The main goal of this study is to establish a correlation between rheologi- cal characterization and ultrasonic application, a non-destructive technique, for assessing the performance of both low and high-voltage transformer oils which deteriorate due to the interplay of several factors. Three materials with vary- ing viscosities (specifically silicone oil, shampoo, and hair gel) were selected to study their mechanical properties using low-frequency rheological measurements and high-frequency ultrasonic techniques. The results obtained revealed three distinct rheological behaviors: Newtonian flow for oils, viscoelastic liquid behav- ior for shampoo and mainly elastic behavior for hair gel, on the one hand, and highlighted the potential of using ultrasound as a complementary tool in the analysis of materials with elastic characteristics, providing information on their mechanical behavior on the other hand.

Keywords: Non-destructive testing, ultrasound, rheology, linear viscoelasticity, soft materials

1 Introduction

One of the major challenges in manufacturing concerns the durability of materials [1, 2]. Beyond the economic implications, reliability during operation, particularly for safety reasons, is of paramount importance [3]. In the case of polymers and oils, degra- dation is especially critical for two main reasons: the widespread use of polymers in everyday life (e.g., textiles, transportation, healthcare) and the high sensitivity of cer- tain polymers to temperature and radiation (UV, IR, etc.) [4–6]. The

specific case of high-voltage transformers clearly illustrates this issue. Due to temperature rises and the possible occurrence of electrical discharges within transformer chambers, the cooling and insulating oil gradually deteriorates, leading to reduced transformer effi- ciency and, in some cases, malfunction [7, 8]. This raises a key question: how can we reliably assess the condition of transformer oils? A particularly practical and attrac- tive approach to address this challenge is the use of ultrasonic measurements. Since these measurements are non-contact, they can potentially be implemented without interrupting operations, allowing continuous or near-continuous monitoring for regular system assessment [9, 10].

However, several fundamental questions must first be addressed. What exactly does the degradation of transformer oils involve? Does it manifest as a change in oil viscosity? If so, does viscosity decrease due to the rupture of polymer chains under the extreme operating conditions within transformers, or does it instead increase, as might be expected, due to a potential crosslinking process occurring over time [11]? In that case, does a cross-linked network actually form, and what would be the charac- teristic size of the resulting aggregates [12, 13]? Other hypotheses are also conceivable. Could there be the formation of particulate inclusions or a gradual incorporation of air bubbles, which would not only modify the rheological properties but also alter the electrical and thermal characteristics of the oils over time [14]?

In the present work, we focus on the first hypothesis, which concerns the evolu- tion of the rheological properties of oils over time. The mechanical behavior of the oils is investigated through both ultrasonic and rheological measurements. The key distinction between these two approaches lies in the measurement frequency [15, 16]. Rheological tests are performed at relatively low frequencies, whereas ultrasonic mea- surements operate at much higher frequencies, offering the major advantage of enabling in-situ assessments and, potentially, real-time monitoring during transformer operation [17, 18].

The question that naturally arises from this observation is how to establish a link between the two types of measurements. To address this issue, we selected model fluids exhibiting well-defined rheological behaviors: a series of silicone oils showing Newtonian behavior with different viscosities, to simulate the viscosity variations of transformer oils over time; a viscoelastic model liquid based on giant micelles (sham- poo), to represent the introduction of moderate elasticity into the system; and a physical gel (hair gel), which exhibits nearly perfect elastic behavior within the linear regime, with a negligible dissipative component [19].

The remainder of this paper is organized as follows. Section 2 describes the experimental setup and procedures employed for both rheological and ultrasonic mea- surements. Section 3 presents and discusses the experimental results, focusing on the comparison between rheological and ultrasonic characterization. Finally, Section 4 summarizes the main conclusions of this study.

2 Experimental details

2.1 Materials

We selected three fluids of distinct natures for the present study:

- A series of polydimethylsiloxane (PDMS) oils, commonly known as silicone oils, chosen for their Newtonian behavior;
- A viscoelastic liquid (shampoo), representing an excellent example of a Maxwell fluid characterized by a single relaxation time;

2.2 A viscoelastic solid (hair gel), whose physical structure provides a high elastic limit, making it particularly suitable for analyzing the flow threshold.Experimental protocol for rheological measurements

Rheological measurements were performed using a TA Instruments AR2000 stress- controlled rheometer. The measurements were carried out with a 40 mm diameter aluminium cone/plate geometry (cone angle: 2 ˚) (Figure 1). Temperature control was maintained by the lower plate using a Peltier element, and the temperature was set to 20 ˚C for all experiments, except for temperature-dependent studies.

Fig. 1 Geometry of the measuring cone.

For all samples, both steady-state flow and dynamic shear measurements were performed. Flow measurements were conducted at a constant temperature close to ambient (20 ˚C) with a shear rate sweep ranging from 0.1 to 1000 s^{-1} in logarith- mic mode, using ten points per decade. Dynamic shear measurements were carried out in two steps: first, identifying the linear viscoelastic region (LVR), and second, determining the frequency-dependent behavior.

In the linear regime defined as the upper stress limit beyond which the storage modulus (G') and the loss modulus (G'') remain independent of the applied stress?the study was conducted for each material before determining the frequency response. Measurements were performed at a frequency of

1 Hz. A logarithmic stress sweep was used between two limiting stresses, σ_{min} and σ_{max}, adjusted according to the material consistency, with seven points per decade.

For the frequency sweep, which represents the most critical part of this type of anal- ysis, the frequency-dependent behavior of each material was determined. The applied stress was set within the previously identified linear regime, and the frequency sweep was performed from 10 Hz down to 0.1 Hz in logarithmic mode, with seven points per decade.

2.3 Experimental protocol for ultrasonic measurements

The experimental setup used for ultrasonic characterization is shown in Figure 2. It consists of a signal generation and reception unit, ultrasonic transducers, a measurement cell, and a digital acquisition system.

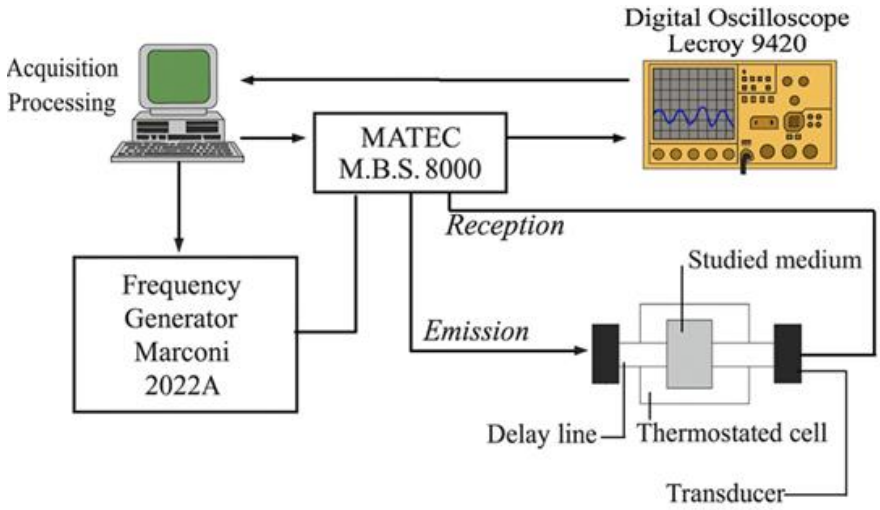

Fig. 2 Experimental setup used for ultrasonic measurements.

A Marconi generator delivers sinusoidal signals that are amplified and shaped into wave trains by the Matec MBS 8000. The recurrence frequency can be adjusted between 10 Hz and 10 kHz, with a pulse width ranging from 0.05 μs to 100 μs. At the generator output, the amplitude can reach up to 1000 V. In the configuration used, the operating frequency extends from 100 kHz to 20 MHz. All control parameters are managed via a computer through an IEEE communication interface linking the various devices.

The resulting electrical wave trains are applied to an ultrasonic transducer which, through the inverse piezoelectric effect, converts them into periodic mechanical waves of low amplitude. The sample under investigation is placed in a stainless-steel measure- ment cell, and all measurements are performed at ambient temperature (approximately 20 °C).

During data acquisition, the investigation frequency is fixed at 10 MHz, correspond- ing to the optimal operating efficiency of the transducer. A portion of the sinusoidal signal obtained is selected for all samples and fitted with a Gaussian function to accu- rately determine the transit time corresponding to the signal maximum. The amplitude is extracted by identifying the overall maximum and minimum of the waveform.

3 Results and discussion

3.1 Rheological measurements

3.1.1 Silicone oils

Figure 3 shows the variation of dynamic viscosity of the silicone oils as a function of shear rate at ambient temperature. All samples exhibit Newtonian behavior over the entire shear rate range, with viscosity remaining independent of shear rate. However, for the two most viscous oils (47V 33000 and 47V 100000), a noticeable decrease in viscosity is observed at high shear rates. This apparent deviation from Newtonian behavior occurs earlier as viscosity increases. Although this could suggest the onset of shear-thinning behavior, it actually results from a measurement artifact. At these elevated shear rates, cavitation occurs within the sample, leading to the expulsion of material from the cone/plate geometry. Consequently, the effective shear stress decreases, producing an apparent drop in viscosity. The Newtonian viscosities of the different oils are summarized in Table 1.

48V 750	**48V 3500**	**47V 33000**	**47V 100000**
0.82 (Pa · s)	3.3 (Pa · s)	30 (Pa · s)	95 (Pa · s)

Table 1 Viscosities of different silicone oils at ambient temperature (20 °C).

3.1.2 Shampoo

Figure 4 presents the variation of dynamic viscosity of the shampoo as a function of shear rate at ambient temperature. The rheological behavior of the shampoo is typical of shear-thinning viscoelastic fluids, exhibiting a Newtonian plateau at low shear rates and a rheofluidizing regime at higher shear rates.

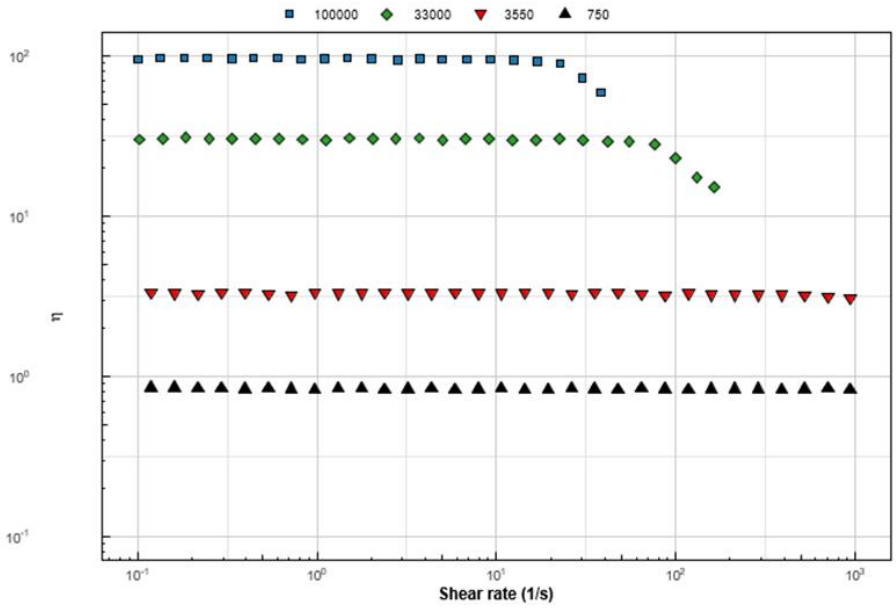

Fig. 3 Viscosity of silicone oils as a function of shear rate at ambient temperature (20 ˚C).

The experimental data were fitted using the Carreau–Yasuda model [20, 21], as shown on the graph. The corresponding fitting parameters are summarized in Table 2, indicating good agreement between the experimental results and the proposed rheological model.

Parameter	Value	Standard error
η_0	1.05259	9.56×10^{-4}
τ	0.05876	1.13×10^{-3}
a	0.85249	1.22×10^{-2}
n	0.14511	9.06×10^{-3}

Table 2 Fitting parameters of the Carreau–Yasuda model for the shampoo at ambient temperature (20 ˚C).

The characteristic relaxation time τ = 0.05876 s corresponds to a shear rate of approximately 17 s^{-1}. This means that beyond this shear rate—which is relatively low compared with those typically encountered during manual hair washing—the shampoo exhibits pronounced shear-thinning, resulting in a marked reduction in viscosity. Such

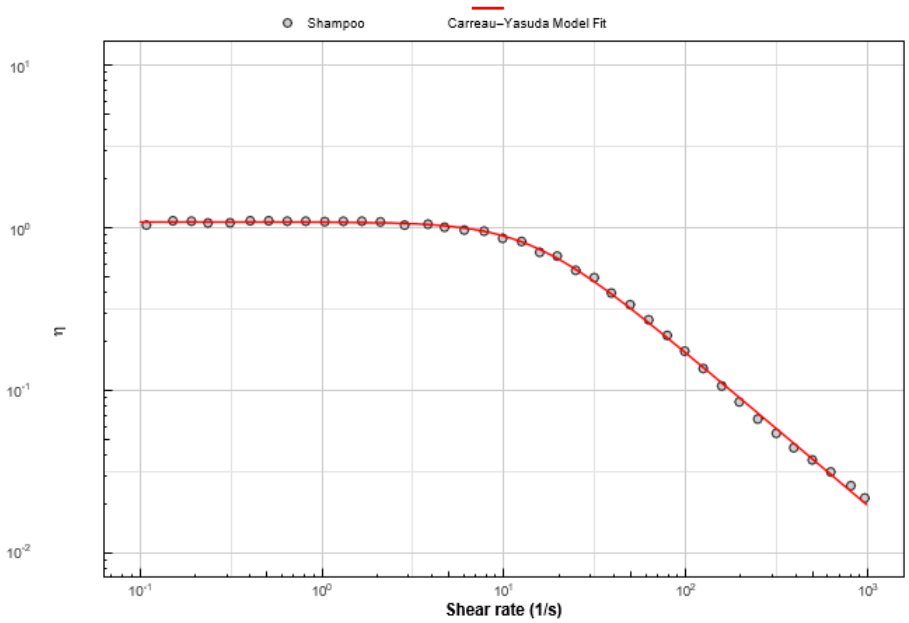

Fig. 4 Experimental data and Carreau–Yasuda model fit for the dynamic viscosity of the shampoo as a function of shear rate at ambient temperature (20 ˚C).

behavior promotes better penetration of the fluid toward the hair roots and enhances the efficiency of deep cleansing within the hair mass. In addition, the value of the flow index n confirms the strong shear sensitivity of this viscoelastic system.

3.1.3 Hair gel

The rheological behavior of the hair gel under shear flow is illustrated in Figure 5. This result reveals the characteristics of a yield-stress (or threshold) fluid, exhibiting a flow initiation stress between 20 and 30 Pa. The pseudoplasticity index is approximately 0.15, which corresponds to an apparent slope of −0.84 on the log–log plot of viscosity η as a function of shear rate $\dot{\gamma}$.

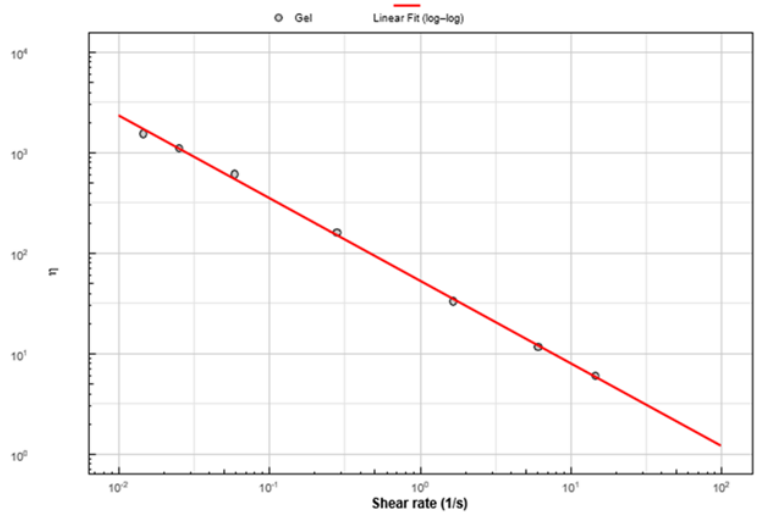

Fig. 5 Flow curve of the hair gel showing the yield-stress behavior.

3.2 Linear domain

Any study of dynamic shear behavior (linear viscoelasticity) must be preceded by the determination of the linear viscoelastic domain (LVD). This preliminary step is illustrated in Figure 6, which presents the evolution of the storage modulus G', the loss modulus G'', and the loss tangent tan δ as functions of the applied shear stress σ and the corresponding strain amplitude γ for the three materials studied: silicone oil, shampoo, and hair gel.

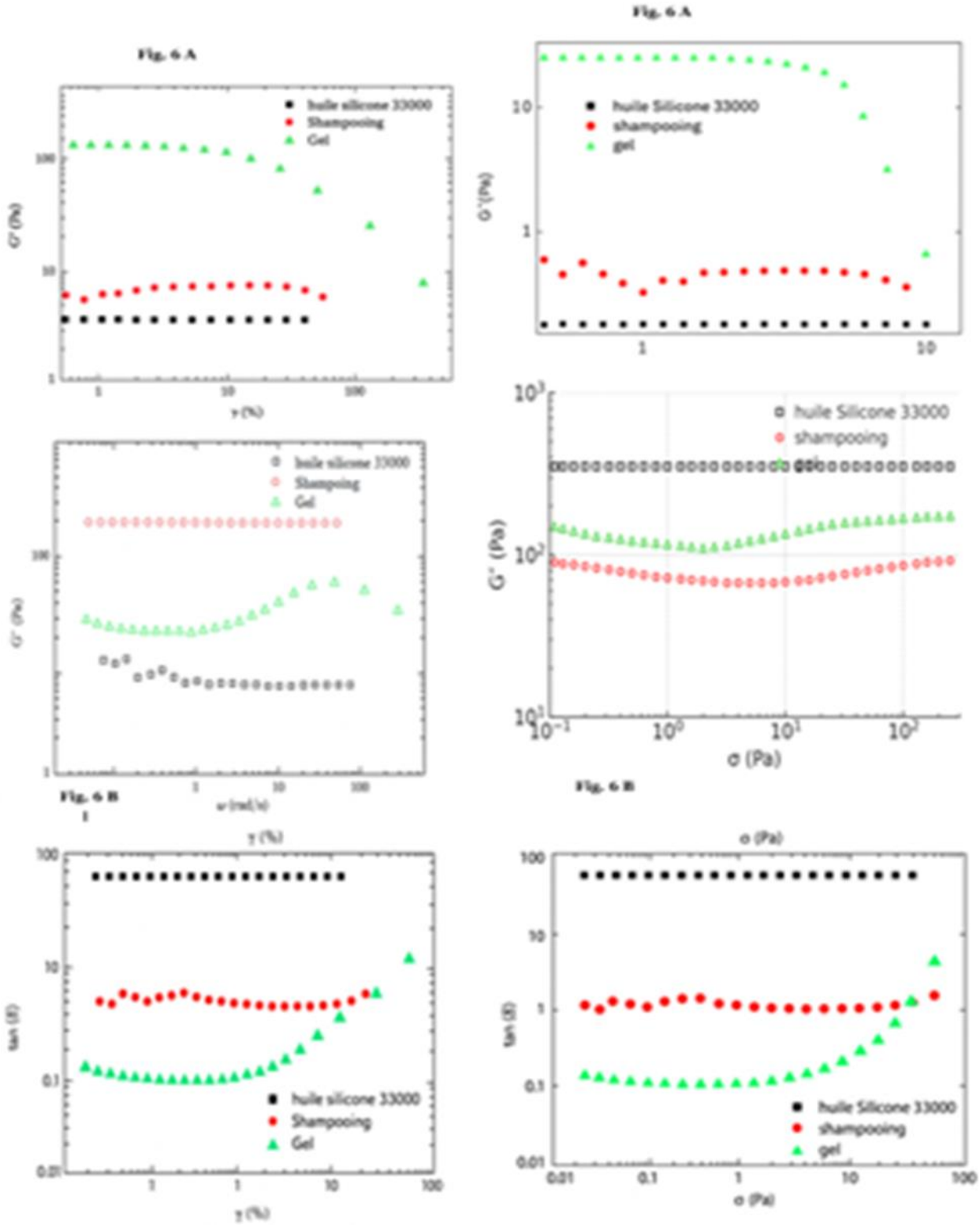

Fig. 6 A.B.C Linear domain

Silicone oil exhibits linear behavior over the entire range of applied stress explored with the rheometer. The shampoo presents an intermediate linear domain, narrower than that of the oil but wider than that of the gel. It is also observed that the storage modulus G' of the shampoo departs from linearity earlier than the loss modulus G''. This deviation from the linear region is accompanied by a decrease in both moduli, consistent with the shear-thinning behavior observed in steady-shear measurements.

The hair gel exhibits the smallest linear domain, where both G' and G'' deviate from linearity at approximately the same strain or stress level. Beyond this domain, G' decreases progressively as the applied stress increases, while G'' reaches a max- imum before decreasing. At the same time, the loss tangent tan δ shows a distinct peak, reflecting the transition from a predominantly elastic to a more viscous response. It is therefore essential to carefully examine the evolution of all viscoelastic parameters before selecting the stress level at which frequency sweep measurements will be performed.

3.3 Ultrasonic measurements

3.3.1 Silicone oils

It should be noted that all silicone oils were tested using ultrasonic measurements, except for the 47V 100000 oil. The very high viscosity of this sample made it par- ticularly difficult to introduce into and clean from the measuring cell. Nevertheless, the results obtained for the other samples indicate that this oil would exhibit similar acoustic behavior.

The ultrasonic propagation velocities and normalized absorption coefficients of the three remaining oils (48V 750, 48V 3500, and 47V 33000) are presented in Figure 7. Within experimental uncertainty, viscosity or molar mass does not appear to significantly influence either the propagation velocity or the absorption in the tested silicone oils. At the reference frequency of 10 MHz, the average propagation velocity and normalized absorption coefficient are approximately 1002.6 $\mathrm{m \cdot s^{-1}}$ and 9×10^{-13} $\mathrm{Np \cdot s^2 \cdot m^{-1}}$, respectively.

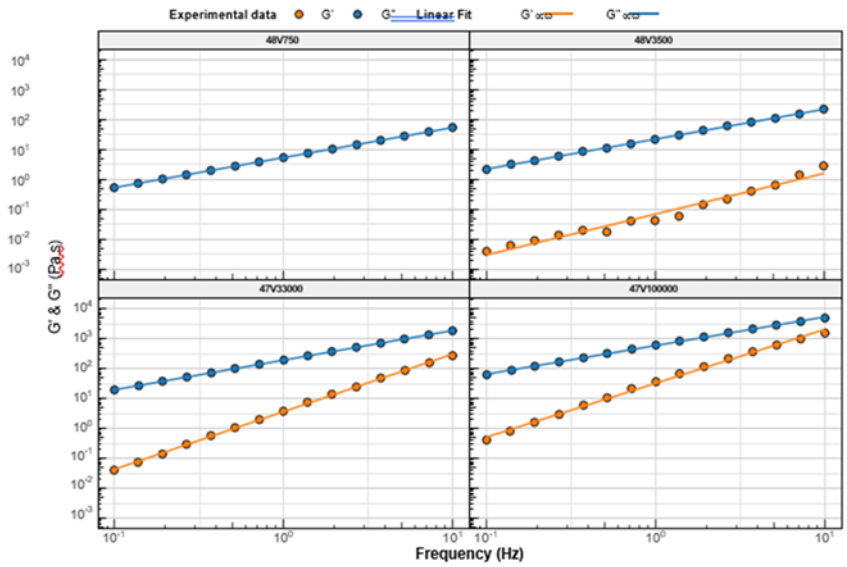

Fig. 7 Dynamic shear moduli (G' and G'') of the silicone oils (48V 750, 48V 3500, 47V 33000, and 47V 100000). At low frequencies, the solid lines represent the asymptotic behaviors $G' \propto \omega$ and $G'' \propto \omega^2$.

3.3.2 Shampoo

Figure 8 shows the dynamic shear moduli (G' and G'') of the shampoo as functions of angular frequency. The behavior is characteristic of a viscoelastic Maxwell fluid: at low frequencies, the loss modulus G'' dominates and increases linearly with ω, while the storage modulus G' varies as ω^2 [22]. Beyond the relaxation frequency, G' and G'' intersect, indicating the transition from a predominantly viscous to a more elastic response.

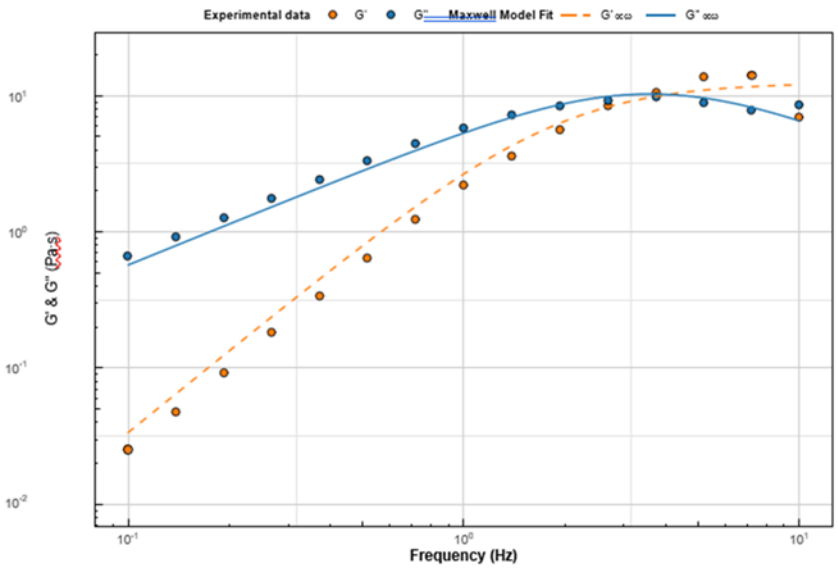

Fig. 8 Dynamic shear moduli (G' and G'') of the shampoo. The solid and dashed curves represent the fits of the data using the Maxwell model with a single relaxation time.

The solid and dashed curves represent the fits of the experimental data using the Maxwell model with a single relaxation time, showing excellent agreement with the theoretical prediction. The Maxwell model is used here because it provides the simplest yet physically meaningful representation of a viscoelastic material, combin- ing an elastic spring and a viscous dashpot connected in series. This configuration allows quantitative interpretation of the characteristic relaxation time $\tau = \eta/G$, which governs the crossover between viscous and elastic behaviors in linear viscoelastic materials.

3.3.3 Hair gel

The measurements for the hair gel are not presented here. The quality of the sam- ple was compromised by the presence of air bubbles, which could not be completely removed and rendered the data unsuitable for reliable analysis.

4 Conclusion

In this work, a comparative study was conducted between rheological and ultrasonic measurements to assess their effectiveness in characterizing materials used in electrical engineering. The results show that ultrasonic measurements present the clear advan- tage of being applicable *in situ* and of operating at very high frequencies. However, these high frequencies correspond to very short wavelengths, which limits the ability to distinguish between oils of similar composition but different viscosities. Conversely, the presence of an elastic component in the material is readily detected by ultra- sonic techniques. Rheological measurements, although requiring sampling, involve only small material quantities and are sensitive enough to differentiate not only materials of similar nature with different viscosities but also the emergence of elastic behavior.

Acknowledgements. The authors would like to express their sincere gratitude to the Energy and Materials Laboratory of the University of Tamanghasset for providing the experimental facilities and technical assistance necessary for this work. They also wish to thank the Editor-in-Chief and the anonymous reviewers for their constructive comments and valuable suggestions, which helped to improve the quality and clarity of the manuscript.

Conflict of interest. The authors have no relevant financial or nonfinancial interests to disclose.

References

[1] Yuan, Q., Shi, C., Jiao, D.: Rheology of Fresh Cement-Based Materials: Fun- damentals,

Measurements, and Applications. CRC Press, London (2022). https:
//doi.org/10.1201/9781003265313

[2] Roussel, N. (ed.): Understanding the Rheology of Concrete. Woodhead Publishing Series in Civil and Structural Engineering. Woodhead Publishing, Cambridge,
UK (2012). https://doi.org/10.1533/9780857095282

[3] Macosko, C.W.: Rheology: Principles, Measurements, and Applications. Wiley- VCH, New York (1994)

[4] Carreau, P.J., De Kee, D.C.R., Chhabra, R.P.: Rheology of Polymeric Systems: Principles and Applications, 2nd edn. Hanser, Carl Hanser Verlag GmbH & Co.
KG (2021). https://doi.org/10.3139/9781569907238

[5] Wilczyński, K.: Rheology in Polymer Processing: Modeling and Simulation. Hanser, Carl Hanser Verlag GmbH & Co. KG (2021). https://doi.org/10.3139/ 9781569906613

[6] Larson, R.G.: The Structure and Rheology of Complex Fluids, 1st edn. Oxford University Press, New York (1999)

[7] Mazzuoli, M., Lacaze, L. (eds.): Physics of Granular Suspensions: Micro- Mechanics of Geophysical Flows. CISM International Centre for Mechanical Sciences. Springer, 6330 Cham, Switzerland (2024). https://doi.org/10.1007/ 978-3-031-55509-1

[8] Ferry, J.D.: Viscoelastic Properties of Polymers, 3rd edn. John Wiley & Sons, Inc., New York, Chichester, Brisbane, Toronto (1980)

[9] Mondal, J., Lakkaraju, R., Ghosh, P., Ashokkumar, M.: Acoustic cavitation- induced shear: a mini-review. Biophysical Reviews **13** (2021) https://doi.org/10. 1007/s12551-021-00896-5

[10] Kuimov, D., Minkin, M., Yurov, A., Lukyanov, A.: Current state of research on the mechanism of cavitation effects in the treatment of liquid petroleum products—review and proposals for further research. Fluids **8**(6) (2023) https:
//doi.org/10.3390/fluids8060172
2024.108678

[11] Hamilton, M.F., Blackstock, D.T. (eds.): Nonlinear Acoustics, 3rd edn. Springer, 6330 Cham, Switzerland (2024). https://doi.org/10.1007/978-3-031-58963-8

[12] Li, J., Guo, J., Dai, Z., Jiang, L., Chen, S.: A hypoelastic dynamic constitutive model to

account for the hysteretic behaviour of soil subjected to cyclic loads. Advances in Civil Engineering **2021**(1), 8873054 (2021) https://doi.org/10.1155/ 2021/8873054

[13] Zhang, J., Shao, Y., Long, Y., He, X., Wu, K., Cai, L., Wu, J., Fang, Y.: Methods for the viscous loss calculation and thermal analysis of oil-filled motors: A review. Energies **17**(18) (2024) https://doi.org/10.3390/en17184659

[14] Myers, D.: Surfactant Science and Technology, 4th edn. John Wiley & Sons, 111 River Street, Hoboken, NJ 07030, USA (2020). https://doi.org/10.1002/ 9781119465829

[15] Baillif, P., Tabellout, M., Emery, J.R.: Dielectric study of the α-relaxation process evolution during polyurethane networks formation. Polymer **41**(14), 5305–5314 (2000) https://doi.org/10.1016/S0032-3861(99)00734-X

[16] Carreau, P.J.: Rheological equations from molecular network theories. Transac- tions of the Society of Rheology **16**, 99–127 (1972) https://doi.org/10.1122/1. 549276

[17] Yasuda, K., Armstrong, R.C., Cohen, R.E.: Shear flow properties of concentrated solutions of linear and star branched polystyrenes. Rheologica Acta **20**, 163–178 (1981) https://doi.org/10.1007/BF01513059

[18] Morrison, F.A.: Understanding Rheology, 1st edn. Oxford University Press, New York (2001)