Title:

Determining the rheological behavior of octadecane as phase change material: First approach.

Authors:

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Abstract

Phase change materials (PCM) are capable of storing thermal energy within a small temperature range due to their high latent heat. In simulations of Thermal Energy Storage (TES) systems with PCMs, natural convection is often not considered, possibly resulting in differences between numerical and experimental results. Since natural convection depends on the viscosity of the melting or melted region, it is important to determine this property in PCMs accurately. This paper presents different measurements carried out with octadecane. Both rotational and oscillatory tests have been carried out and the influence of the gap and the heating and cooling rate on the results has been analyzed. A first approach to the measurement methodology for obtaining the viscosity of octadecane during phase transition and melting is proposed, using a plate as geometry and a Peltier plate as temperature controller.

Keywords: Viscosity, Rheology, Natural convection, Phase Change Materials

Nomenclature

c	multiplier fitting coefficient (-)
f	frequency (Hz)
G'	elastic module (Pa)
G''	loss module (Pa)
k	thermal conductivity (W/(m^2·ºC))
n	exponent fitting coefficient (-)
Ra	Rayleigh number (-)
T	temperature (ºC)

Greek symbols

η	steady shear viscosity (Pa·s)
$\dot{\gamma}$	shear rate (1/s)
ω	angular frequency (rad/s)
η^*	complex viscosity (Pa·s)
t	shear stress (Pa)

Subscripts

e	effective
m	melting

Abbreviations

DSC	differential scanning calorimetry
PCM	phase change material
TES	thermal energy storage

1. Introduction

The importance of the rational use of energy, the promotion of energetic saving and the reduction of greenhouse gases have led to an increase in research into renewable energies and Thermal Energy Storage (TES). Exergy demand for space heating and cooling of buildings is very low, since a room temperature about 20ºC is very close to the ambient conditions. However, energy sources like fossil fuels are commonly used to satisfy these small exergy demands. TES allows the use of low value energy sources whose supply fluctuates over time, in these low exergy systems.

Through an appropriate TES system, the energetic flow can be adjusted to a constant or variable energy demand. The TES technique based on the use of PCMs has aroused great interest as a consequence of the possibility of high energy storage density in a narrow temperature range and because the absorption/release of energy is carried out at a constant temperature or within a narrow temperature range. Research done in recent years has led to an increase in the number of available PCMs as well as knowledge of their behavior, as shows the reviews with a high number of references presented in this field [1-3].

The incorporation of PCMs in a particular application usually requires a numerical analysis that allows optimization of the system performance. In current models, the natural convection in the PCM is not usually considered. However, various experimental works have shown that this term must be taken into account.

When there is not a forced movement in a fluid, a spontaneous movement in the fluid may appear (and therefore convective phenomena) if there are density gradients (due to temperatures gradients) under a gravitational field. Apart from the buoyancy forces, this convective phenomenon called natural convection will depend on viscous forces, that will prevent that movement. For this reason, the determination of the viscosity property is relevant. In TES systems, where PCM is in bulk form or macroencapsulated (no forced movement of the PCM), the natural convection could take place in the liquid phase or during the transition. Natural convection will be more relevant in the melted phase than in the melting phase, due to the increasing values of viscosity during the phase transition.

More specifically in macroencapsulated PCM or in the bulk PCM, during melting, heat is transferred to the PCM first by conduction, and later by natural convection, because the thickness of the liquid region increases near the heat transfer surface. Due to the lower themal conductivity of liquid PCM in comparison to solid PCM, the heat transfer by conduction almost becomes negligible when the melting continues, dominating the natural convection in the liquid phase. On the other hand, during solidification, the heat transfer phenomenon is dominated by conduction. In this case, natural convection is important at the beginning and as the time goes, natural convection become almost zero compared to the conduction. The review of Jegadheeswaran and Pohekar [4] compiles many experimental references that prove this phenomenon of natural convection in latent thermal energy storage systems.

In view of the presence of the natural convection phenomenon in TES systems with PCM, and the dependence of the natural convection on the viscosity, this property should be measured. How accurate the viscosity must be determined, it will depend on the specific application, and it would have to be calculated according to an uncertainty analysis.

Arkar and Medved [5] pointed out the importance of determining PCM properties. They compared the results of their numerical model with experimental results. The comparison confirmed their hypothesis of the importance of the role played by the thermal properties of the PCM, especially in slow running processes. Specifically, they studied the influence of the Heat capacity-Temperature curve obtained with a DSC for different heating and cooling rates on the results of a TES system with PCM spheres. The first studies to take natural convection into account were those of Sparrow et al. [6] and Bathelt et al. [7]. To simulate this heat transfer mechanism, some authors [8-10] considered an effective thermal conductivity, whose value will depend on the viscosity property:

$$\frac{k_e}{k} = c \cdot Ra^n \quad (1)$$

Costa et al. [11] studied numerically the thermal behavior of three PCMs, a paraffin (octadecane) and two metals, confined in a rectangular domain where the natural convection in the fluid and the conduction in the solid were both considered. In the case of the octadecane, the authors pointed out a bad fitting with the experimental results in the upper part. The PCM started to melt at the sides. This melted PCM occupied the upper part of the rectangular domain as a consequence of its lower density, the melting in this zone being faster. The authors think that the discrepancies between the theoretical and experimental results were caused by thermal inertias, systems instabilities, thermal losses, lack of reliable information about the physical properties of the materials, 3D behavior, consideration of constant thermophysical properties, density variations, high calculation time and an important change in the viscosity with the temperature. The same authors in a later publication [12] observed discrepancies between their numerical solution and experimental results. These discrepancies could be attributed to the viscosity value taken in the numerical simulation, a constant value of 0,003898 Pa·s. To check its influence, they took the value of viscosity at a higher temperature. The change in the viscosity value caused differences in the melting front. However, the variation of viscosity with temperature did not explain other differences.

Due to the dependence of natural convection on the Rayleigh number and therefore on viscosity. It is therefore necessary to determine rheological behaviour, and specifically the viscosity property dependent on the temperature and even in the phase transition, in order to incorporate these data in natural convection simulations. However, there are few reported studies about the rheological properties of PCMs.

In fact, a recent review about the methods of characterization of PCMs, accomplished in the field of the COST Action TU 0802, proves that there is a lack of researchers working in the rheological behavior of PCMs within this framework [13].

Tipvarakarnkoon et al. [14] analyzed the rheological properties of three commercial coconut fats and undertook a structure analysis during their solid-liquid and liquid-solid phase transition. They obtained flow curves (viscosity vs. shear rate) in the liquid phase, using a double gap rotational cylinder. The shear rate was increased from 100 to 1500 1/s in 2 minutes and then decreased from 1500 to 100 1/s in 2 minutes. The data was collected every 4 seconds at each state measurement. To evaluate the phase transition, the G’ and G’’ modulus were obtained from oscillatory tests. Specifically, stepwise temperature sweeps were carried out with a cooling rate of 0.5 K/min. Values of tan G’’/G’>>1 corresponded with the liquid phase, values of tan G’’/G’<<1 corresponded with the solid phase and values of tan G’’/G’=1 corresponded with the phase transition.

The work presented here is part of the COST Action TU0802 (Next generation cost effective phase change materials for increased energy efficiency in renewable energy systems in buildings). One of the objectives of this action is to develop standardized methodologies to characterize PCM, in which the importance of rheology for PCM is also identified [13]. This work also represents a contribution to Task 42-Annex 24 of the International Energy Agency (IEA) [15]. Specifically, it is included within the development of measuring and testing procedures to characterize new storage materials reliably and reproducibly. The aim of the work presented here is to develop a measurement procedure of the viscosity property. In Task 42-Annex 24, much work has already been accomplished on the standardization procedure for Enthalpy-Temperatures curves with Differential Scanning Calorimetry. Octadecane has been used for this, and is also used here for viscosity property measurements.

2. Material and methods

2.1 Materials

According to the chemical nature of the material, PCMs can be classified as organic or inorganic. In the present work, an organic material will be characterized. Although most organic commercial PCMs are blends of different alcanes, octadecane with a purity of 98.11% was chosen as the first test sample. The octadecane was purchased from the Sasol company, specifically the product Parafol® 18-97 [16]. Table 1 shows the octadecane properties [17].

2.2 Apparatus and instrumentation

The measurements presented were made with a Haake Mars II control stress rheometer supplied by Thermo Scientific. The sample temperature was controlled using a Peltier plate. A titanium plate with a diameter of 60 mm was used for the rheological characterization of the PCMs opposite to the double gap cylinder used by Tipvarakarnkoon et al. [14] which probably would give different results. In these geometries, lower shear stresses can be achieved due to the higher surface, being appropriate to measure fluids with low viscosity, such as the melted octadecane. However this geometry includes approximations when calculating the shear stress and shear rate, as the plate geometry.

2.3 Method

Two types of tests and three replications of each test were conducted to analyze the rheological behaviour of the PCM: 1) rotational test and 2) oscillatory tests.

The Viscosity-Shear rate curves or rotational tests were obtained through a shear rate sweep from 0.001 1/s to 1000 1/s. For this purpose, stress was applied to the sample. The measurement of the viscosity was accomplished when the material reached the steady state. The stress was increased logarithmically and the process was repeated, providing the flow viscosity curve. The steady state was reached when the variation of the applied stress varied less than 1% during 40 seconds, with a maximum time of 60 seconds.

Three types of oscillatory test were carried out: strain or stress sweeps, frequency sweeps and temperature sweeps. The frequency sweeps here presented cover a frequency range from 0.01 Hz to 1 Hz and were carried out within the viscoelastic region. The viscoelastic region covers the values range of strain or stress where the relationship stress/strain is linear. Stress sweeps were previously carried out from 0.01 to 100 Pa at a frequency of 1 Hz. The permanence within the viscoelastic region has to be guaranteed above 1 Hz. This 1 Hz frequency is considered sufficient since above 1 Hz the measurements may present considerable inertia. In spite of the correction of the software, it is advisable not to take into account measurements at high frequencies. Besides, in this analysis measurements at low frequencies are of interest since the PCM is at rest.

In spite of accomplishing three replications of each experiment, just one result is shown. Small differences among the three replications were observed, always within an acceptable tolerance. Slight changes in some of the experiment parameters were considered in the evaluation of these replications. Specifically the gap parameter was considered. The gap is the distance between the Peltier plate and the geometry, which determines the amount of sample.

3. Results

Since the PCM is at rest in the TES system, a rotational sweep was initially considered for determining the viscosity at very low shear rates. However, this approach was quickly rejected because it was observed during the tests that when the geometry turns at a specified velocity, it may cause the crystals formed to break, influencing the results in this mode (this is only one of the phenomena that could cause erroneous measurements in rotational experiments). Measurements in oscillatory mode were thus proposed. First of all, a stress or strain sweep has to be undertaken to determine the linear viscoelastic region of the PCM, in this case octadecane. This region has to be determined for different frequencies and for different temperatures. Regarding to the frecuencies, this must not be too high, to avoid inertia problems, and considering that lower frequencies mean higher linear viscoelastic region. The range of interest relates to temperatures within the phase transition and in the liquid state. Figure 1 shows the results derived from these stress sweeps at 1 Hz. At 29°C the G’ module is not visible in the graph because the PCM is completely melted and its elastic part is too low for the rheometer to obtain a value. At 27.6°C (during the phase transition), it can observed that at very low stresses (up to 1 Pa approximately) the measurements show considerable noise. From liquid to solid, when the transition takes place, the material starts to form molecular bonds, what it means an abrupt increase in the G’ module due to the fast transition, leading to an increase in the complex viscosity (see figure 2 to support this fact). With high values of viscosity, low stresses will cause even lower strains. These strain values that are going to be measured by the sensor of the rheometer will be low, they might be close to the resolution of the displacement sensor of the rheometer, 12 nrad in this case. If the values obtained from measurement with low stresses are discarded, it can be seen that the linear viscoelastic region reaches values up to 100 Pa.

Once the linear viscoelastic region was determined, the “oscillatory temperature steps” procedure was carried out both for the melting curve and for the solidification curve. The procedure was done using temperature steps of 0.1ºC. Since the sample needs an equilibrium time to reach the Peltier plate temperature, a stabilization time of 60 seconds was chosen. The sample was loaded at its liquid phase. The gap, which is the distance between the Peltier plate and the geometry, was adjusted so that the sample formed a meniscus. Once the sample was placed between the Peltier plate and the geometry, the value of the normal force present was fixed as a reference. When the PCM changed phase, the rheometer thus adapted the gap between the Peltier plate and the geometry in such a way as to maintain the same normal force value, since with the phase transition the sample changes its volume and this would affect the normal force. For this reason, plate geometry was chosen in order to be able to adapt the gap. Figure 2 shows the obtained results. According to this graph, the octadecane changes from liquid to solid at 27.3-27.4°C and from solid to liquid in the temperature range 27.6-28°C. Figure 3 shows how the rheometer changed the gap during the phase transition to maintain the same normal force reference value.

Frequency sweeps at different temperatures were also carried out. The obtained values (figure 4) at very low frequencies (corresponding to materials at rest, about 0.01 Hz or lower) are the same as at higher frequencies (1 Hz) that it could mean a Newtonian behaviour.

To know if the values of complex viscosity can be extrapolated as values of steady shear viscosity, since the phase transition can not be measured from rotational tests, it is necessary to compare in the liquid phase the curve in steady state flow η-$\dot{\gamma}$ to the curve of the frequency sweep η*-ω. If these values fit well, the values of complex viscosity will be able to be taken during the phase transition as shear viscosity values, as if the steady state flow curve had been obtained. This relation is known as the Cox-Merz rule [18]. Both curves are shown in figure 5. It can be observed that the steady state flow curves show a viscosity value of about 4.0 mPas, when the shear rate value is greater than 5 1/s. The measurements at a shear rate lower than 5 1/s show considerable noise. The frequency sweep curves show a complex viscosity value of about 4.4 mPas below an angular frequency of 5 rad/s. Both values (4.0 and 4.4 mPas) are very close, but they can not be compared over the whole range because of the noise. In the frequency sweep, it is possible that above values of 5 rad/s there are problems of inertia that the rheometer cannot correct, because the rheometer software cannot obtain an inertia parameter during measurements. High angular frequency means inertia problems, where inertia is related quadratically to the angular frequency. In the case of the steady state flow curve, the problem of noise in the measurements at low shear rates is probably due to the minimum torque that the rheometer can execute and also the low viscosity of the melted octadecane. Low shear rates will mean low stresses when the material is melted due to its low viscosity. Low stresses will mean low torque applied by the rheometer, close to the minimum torque that the rheometer can execute.The rheometer user manual advises working with torque values ten times above the minimum torque of the equipment (0.05 μNm). Figure 6 shows the torque applied by the rheometer during the experiments and it can be observed that the noise zone corresponds with torques below 0.5 μNm. So, if the noise of both rotational and oscillatory curves is not taken into account and attention is focused on the intermediate range (approximately from 1 to 10 rad/s or 1/s), a correspondence between the η-$\dot{\gamma}$ and the η*-ω curve is observed and therefore it can be stated that the Cox-Merz rule is fulfilled. In this way, the values of complex viscosity obtained in oscillatory tests will be able to be taken as values of shear viscosity, as if the flow curve had been obtained. It is important to guarantee that any transient effects due to the load of the sample, inertia or thixotropy behaviour is influencing the measurement. For example, in structured fluids, their load on the Peltier plate can cause the partial rupture of the structure. In these cases it is important to give a time so that the sample recovers the structure. It is also important to use geometries for which the viscosity calculation is exact. This normally means cone-plate, because the shear rate is constant over the whole radius of the geometry.In this case, plate-plate has been used since the normal force must be controlled, due to the volume changes during the phase transition. The observed differences between the two curves may be due to the approximations in the calculations to obtain the viscosity value with the plate-plate geometry, since there is a shear rates gradient from the center of the plate up to the edge.

3.1 Influence of the gap and of the heating and cooling rate on the measurement of the viscosity property

As the gap between the lower and the upper plate can be chosen by the operator, its influence on the results has been studied. The influence of the heating and cooling rate has also been analysed. These factors must be taken into account due to the gradient between the sample temperature and the room temperature, and due to the low thermal conductivity of PCMs. A gap of 1.4 and 0.4 mm was investigated. Regarding the heating and cooling rates, rates from 2 to 0.1°C/min were tested. Figures 7 and 8 show the complex viscosity values obtained for both heating and cooling rates, and for the high and low gap respectively. A higher apparent hysteresis between the melting and the solidification curves as consequence of the method (that is to say, due to the test conditions: heating and cooling rate and sample size) and not due to the hysteresis as a material property, can be observed when working with higher gaps and with higher heating and cooling rates. The solidification temperature is displaced to lower temperatures because of the bigger size of the sample. Since the geometry at the beginning of the experiment is at room temperature, the octadecane will start solidifying in a first layer on the Peltier plate. In this way, for the same cooling rate, it will take a longer time for the geometry to note a solidified layer under itself. These differences are not so notable for the melting curves, perhaps because in this case the lower part will be at a higher temperature, helping the natural convection in the sample. As expected, the hysteresis increases with higher heating and cooling rates due to the lack of thermal equilibrium between the sample and the Peltier plate.

4. Discussion

The inertia problem mentioned above is especially significant when working with low viscosity materials such as octadecane in its melted phase. The rheometer software does not provide any parameter that gives an idea of the inertia presented in the measurements. To ease the inertia, a plate made of titanium was chosen, although the rheometer motor has the biggest load of the inertia in the total system. It may also be of interest to work with lower frequencies since the relation of the inertia with the frequency is quadratic.

During the experimental measurements, different complex viscosity results were observed when the tests were carried out with different stresses (from 0.1 to 10 Pa) in spite of being within the linear viscoelastic region defined in figure 1. According to figure 9 and 10, higher stress values caused the octadecane phase transition (melting) to start at a lower temperature. One hypothesis is that a very high stress level may cause a rupture between the lower layer (partially liquid) and the upper layer (solid), this latter layer adhering to the geometry. The geometry thus starts to turn without encountering high resistance and it seems that the sample is in its liquid state when there are still crystals. In the same manner, a possible influence of the frequency on the results has been analysed. It was observed in previous sections that octadecane (in its liquid state and during phase transition) could be considered as a Newtonian fluid (since the Cox-Merz rule was fulfilled). Accordingly, in principle the value of the set frequency in the oscillatory temperature ramps should not affect the results (whenever there is little inertia). It is possible that the energy associated to high frequencies may increase the temperature of the sample. However, the results obtained do not show any clear evidence for this hypothesis (figure 11).

5. Conclusions

According to the results of the tests here presented, a first approach to obtain viscosity values of PCMs during their liquid state and during phase transition is proposed. These values will be able to be used in numerical simulations of latent thermal energy storage systems, if natural convection plays an important role in the heat transfer phenomenom.

Tests in oscillatory mode must be carried out. The steps to be executed with a plate as geometry and a Peltier plate as temperature controller are as follows:

1) Strain or stress sweep at different temperatures (both in the liquid phase and during the phase transition). A frequency about 1 Hz is advisable. With this step, the linear viscoelastic region will be determined. With melted PCM, lower frequencies should perhaps be used.
2) Frequency sweeps in the molten and in the phase transition region. The frequency sweep must be carried out at a stress or strain within the linear viscoelastic region (defined previously in step 1). In any case, it would not be advisable to use high stress or strain as this could affect the measurements. It is important to work far from the minimum angular displacement value of the rheometer. This frequency sweep will provide information about the rheological behaviour of the sample in the molten and in the phase transition region (Newtonian or non-Newtonian, if the Cox-Merz rule is fulfilled). If the sample is Newtonian, step 3 may be tested at any frequency. Frequencies below 1 Hz are recommended to avoid problems of inertia in the case of measuring with control stress rheometers, especially with melted PCM.
3) Once the linear viscoelastic region is determined and the frequency test executed at different temperatures, an oscillatory temperature ramp test (or by steps) must be executed (both for melting and solidification). If the PCM is non-Newtonian, the test will be carried out at 0.01 Hz and if it is Newtonian, 1 Hz will be sufficient. Low gaps must be used to avoid temperature gradients in the sample. Different gaps should be tested. When the results do not vary, an appropriate gap will have been found. Different heating and cooling rates should be tested, and the rate for which the Complex viscosity-Temperature curve does not change should be selected. In addition, the normal force during the test must be controlled. This must be always the same. The rheometer will adapt the gap as a consequence of the volume change during the phase transition.
4) To know if the complex viscosity values can be extrapolated as shear viscosity values, since the phase transition cannot be measured with steady state flow, the curve in steady state flow η-$\dot{\gamma}$ and the curve of the frequency sweep η^*-ω in the liquid state must be compared. If these values fit within the uncertainty range previously defined from a viscosity standard of the same order of magnitude of the PCM to analyze, the complex viscosity values will be taken during the phase transition as shear viscosity values, as if the steady state flow curve had been obtained (Cox-Merz rule). If these curves do not fit well, the complex viscosity values may not be considered as shear viscosity values.

Notes about the methodology: 1) The procedure described above is for working with plate geometry and a Peltier controller. The plate is not necessarily the most appropriate geometry, due to the shear gradient across the sample. However, a cone does not allow the normal force to be controlled since the gap is set by the geometry itself. Regarding the temperature controller, it would be interesting to use an “environmental test chamber” in order to provide more realistic results for PCMs with phase transition temperatures well above or below room temperature, since this controller would avoid the temperature gradient in the sample. However, the air flow in the test chamber can cause the melted PCM to leave the geometry due to its low viscosity. Also, the problem may be solved with the combination of a Peltier Plate and an upper heated plate. 2) If the PCM during its phase transition or in its liquid phase is not a Newtonian fluid, or if the measurements with the tested frequency present considerable inertia, it is necessary to test the oscillatory temperature ramp at very low frequencies, about 0.01 Hz (PCM at rest). The problem in this case is that the rheometer needs at least one period to obtain a value of complex viscosity. If the frequency is 0.01 Hz, the rheometer would need at least 100 seconds. If the heating and cooling rate is, for example, 0.2°C/min, in these 100 seconds the temperature will vary by about 0.34°C. The measurement would not therefore be a correct measurement due to the inconstant temperature. In this case, precise measurements at set temperatures should be carried out. For this reason, a temperature steps procedure is considered to be better than a temperature ramp.

Acknowledgments

This work was carried out by Mónica Delgado during a research stay at the Fraunhofer ISE Institute. Special thanks are extended to this research centre for the support provided. The work was developed within the framework of the research project ENE2008-06687-C02-02 and funded by a grant from the CAI Programa Europa and by a STSM grant from the COST Action TU0802.

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Figures and tables captions

Figure 1. Stress sweeps at different temperatures (melted phase and phase transition). Frequency=1 Hz. Gap~0.5 mm.
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Figure 4. Frequency sweep at 29 and 27.6ºC. Shear stress=1 Pa. Gap~0.5 mm.
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Figure 9. Influence of the applied stress (within the linear viscoelastic region) on the Complex viscosity-Temperature curves. Gap=1.4 mm. Heating rate=0.5°C/min. Frequency=1 Hz
Figure 10. Influence of the applied stress (within the linear viscoelastic region) on the Complex viscosity-Temperature curves. Gap=0.4 mm. Heating rate=0.5°C/min. Frequency=1 Hz
Figure 11. Influence of the frequency on the Complex viscosity-Temperature curves. Shear stress=1 Pa. Gap=0.5 mm. Heating rate=0.5°C/min.
Table 1. Properties of the used material [17]

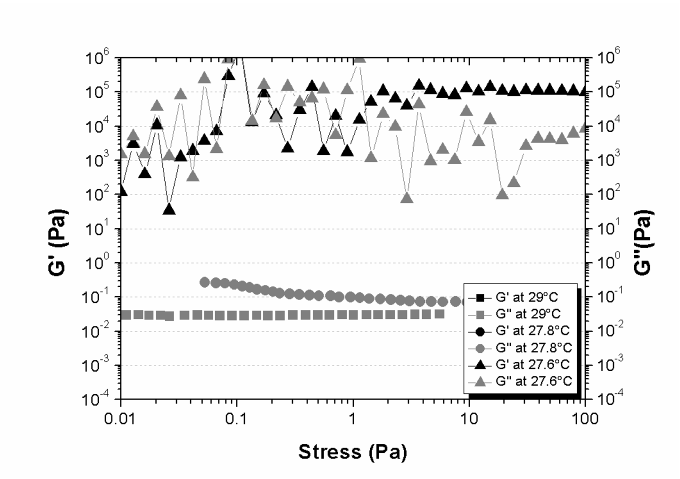


Figure 1.

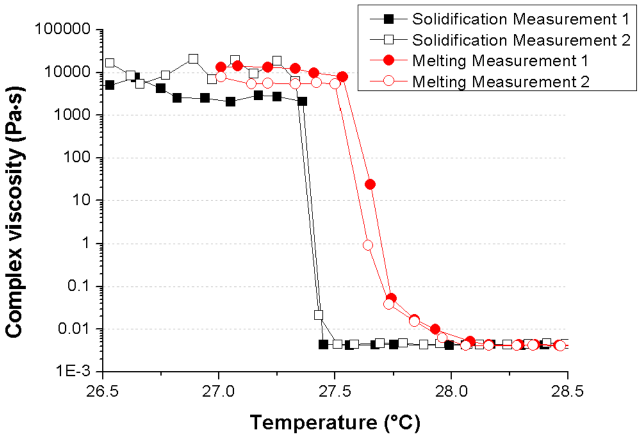
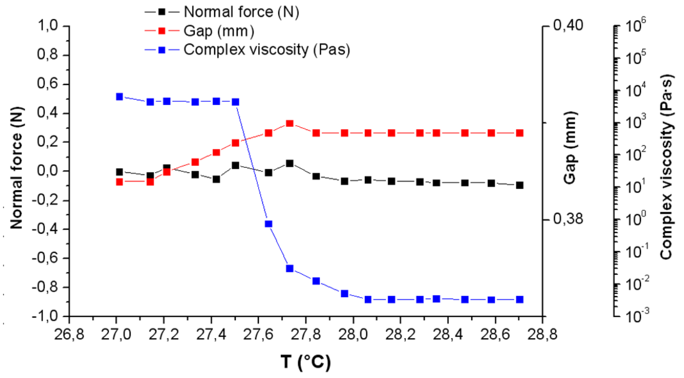


Figure 2.



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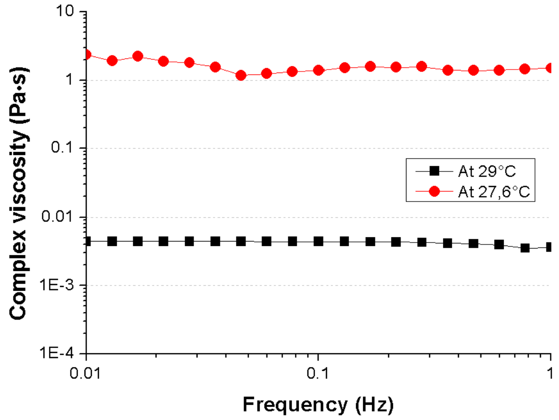


Figure 4.

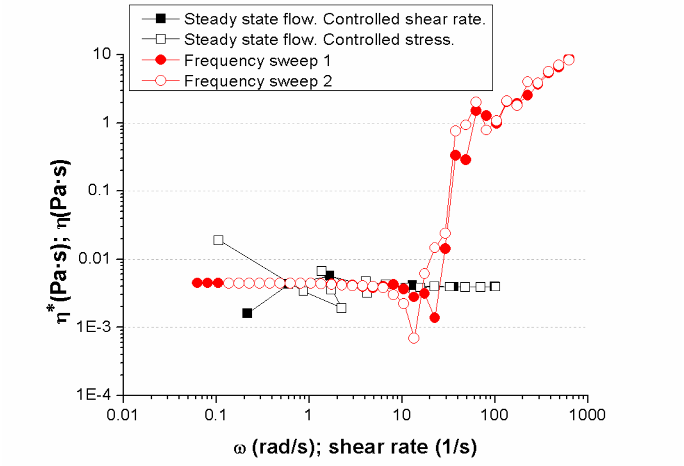
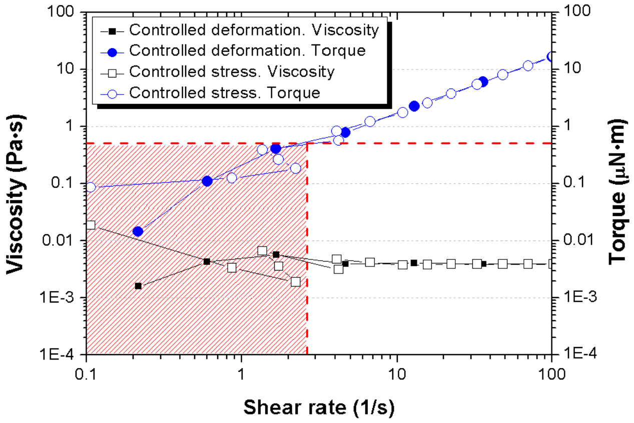


Figure 5.



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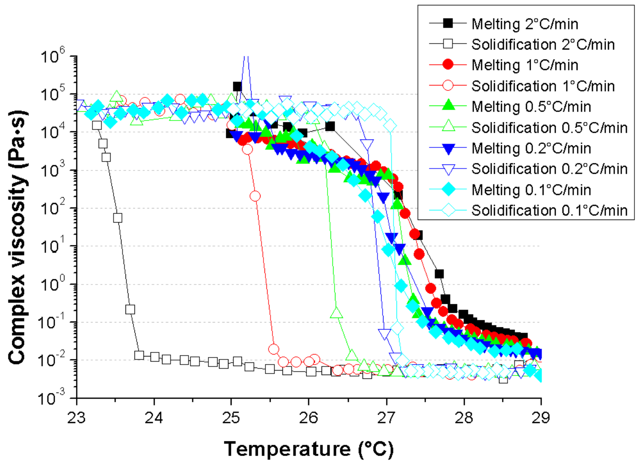

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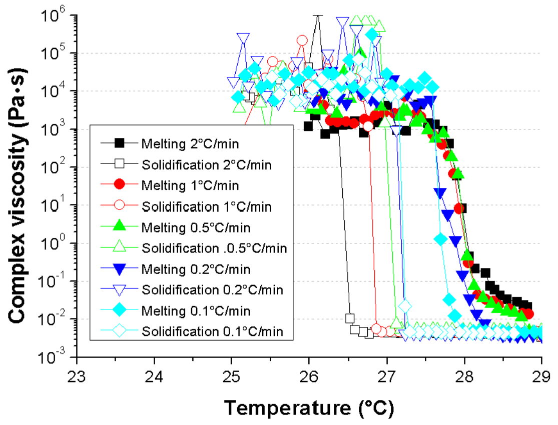

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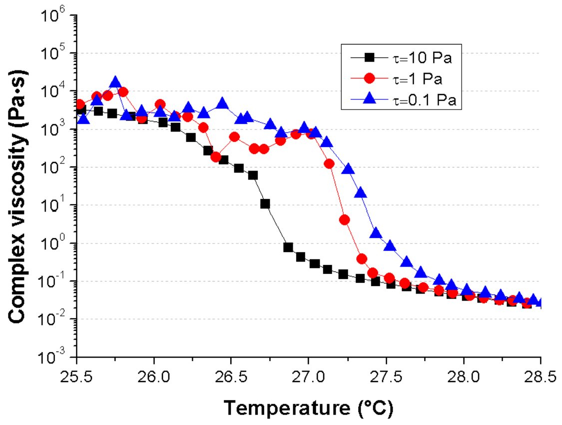

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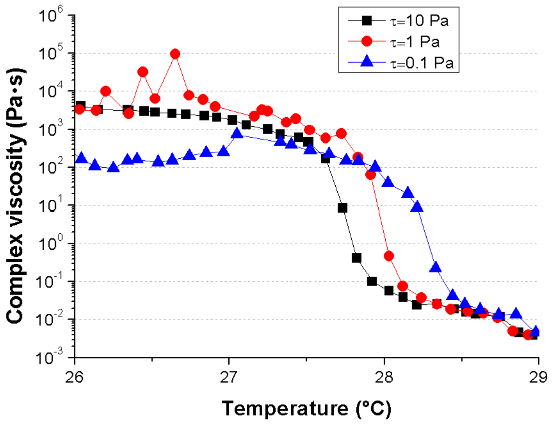

Figure 10.

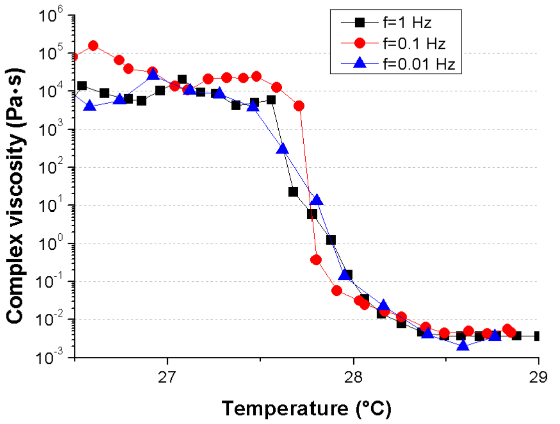

Figure 11.

Purity of the used sample	98.11%
Phase change temperature Tm (ºC)	27.85
Phase change enthalpy Δh (J·g^{-1})	241.66
Specific heat solid at 298,15 K Cp (J·g^{-1}·K^{-1})	1.91
Specific heat liquid at 325 K Cp (J·g^{-1}·K^{-1})	2.23

Table 1.