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Research Article

COMPARATIVE ANALYSIS OF PHASE TRANSITION TEMPERATURES AND RHEOLOGICAL CHARACTERISTICS OF *p-n*-ALKYL- AND *p-n*-ALKYLOXY-BENZOIC ACIDS

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p-n-Alkyl- and *p-n*-alkyloxy-benzoic acids are two prominent classes of liquid crystal compounds with diverse applications in various fields. In this study, we conduct a comparative analysis of our prior research focusing on the phase transition temperatures and rheological characteristics of these two prominent classes of liquid crystal compounds. By this, we aim to discern any notable similarities or differences in their rheological profiles. Analysis of shear stress, angular frequency, and shear rate reveals interesting differences in the observed response of these materials. The presence of oxygen in *p-n*-alkyloxybenzoic acids (*n*OBA) elevates the temperatures at which phase transitions occurs. Moreover, owing to their non-planar structure, *p-n*-alkyl benzoic acids (*n*BA) exhibit greater viscoelasticity compared to *p-n*-alkyloxybenzoic acids *(n*OBA) acids.

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СРАВНИТЕЛЬНЫЙ АНАЛИЗ ТЕМПЕРАТУР ФАЗОВЫХ ПЕРЕХОДОВ И РЕОЛОГИЧЕСКИХ ХАРАКТЕРИСТИК *p-n*-АЛКИЛ- И *p-n*-АЛКИЛОКСИ-БЕНЗОЙНЫХ КИСЛОТ

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А Н Н О Т А Ц И Я

p-n-Алкил- и *p-n*-алкилокси-бензойные кислоты – это два известных класса жидкокристаллических соединений с разнообразным применением в различных областях. В этой статье мы проводим сравнительный анализ наших предыдущих исследований, уделяя особое внимание температурам фазового перехода и реологическим характеристикам этих двух известных классов жидкокристаллических соединений. Таким образом, мы стремимся выявить любые заметные сходства или различия в их реологических профилях. Анализ напряжения сдвига, угловой частоты и скорости сдвига выявляет интересные различия в наблюдаемой реакции этих материалов. Присутствие кислорода в *p-n*-алкилоксибензойных кислотах (*n*OBA) повышает температуры фазовых переходов. Более того, из-за их более высокого соотношения сторон и неплоской структуры *p-n*-алкилбензойные кислоты (*n*BA) проявляют большую вязкоупругость по сравнению с *n*OBA кислотами.

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Introduction

Many materials go through a transition from a structured crystalline state to a uniform liquid phase. But certain substances deviate from this norm, transforming into fluids with directional properties. These ordered liquids occupy a unique state, which is an intermediate between a solid crystalline phase and an isotropic fluid phase, denoted as a liquid crystal (LC) phase or mesophase. LCs are the core technology behind the displays of most smartphones, computers, televisions and other devices. They are also employed in several other intriguing applications. Recent studies have emphasized the critical role of liquid crystals in the development of advanced holographic plastics [1]. The use of liquid crystals represents a major technological advancement, enabling the production of thicker and more diverse structures with superior mechanical properties [2]. Eventually, thermotropic liquid crystals have become the most technologically important materials as they find applications in various fields due to their unique properties. They are used in displays, optical devices, biomedical imaging, smart windows, sensors, photonics, cosmetics and lubrications, microwave components and devices, constant current device and in diagnosis of cancer [3–7]. Two of the most notable candidates among these are *p*-*n*-alkylbenzoic acid and *p*-*n*-alkyloxybenzoic acid mesogens (from now on, they will collectively be referred to as *p*-*n*-alkyl(oxy)benzoic acids). These mesogens have unique molecular structures and intrinsic qualities (Fig. 1). These compounds find utility as intermediates in synthesizing liquid crystal materials [8–11] substances of biological importance [12], and as standalone research materials [13].

In material science, rheology plays a critical role in unlocking the secrets of liquid crystals. This field of study analyzes how materials flow and deform under stress. The flow behaviour of complex fluids is hard to understand. By employing techniques like oscillator and steady shear, rheology provides valuable insights into how liquid crystal molecules align themselves under various flow conditions [14]. Changes in microstructure directly impact the rheological properties of the liquid crystal. Understanding this interplay between flow and internal structure is essential for predicting the behaviour of liquid crystals and developing new applications, ranging from lubricants to coatings [15–17].

The study of liquid crystal rheology is a fascinating aspect of condensed matter physics, as it explores the flow behaviour and viscoelastic properties of materials that exhibit both liquid-like and crystalline characteristics. In a prior work, we characterized the rheological characteristics of *n*OBA compounds [18, 19]. Here, we present a comparative analysis of these rheological measurements with the results from our earlier study on the *n*BA series [20]. Figures 2–6 replicate the data and presentation style of corresponding figures from earlier publications [18–20]. This comparative analysis aims to elucidate how the presence of ether linkages and the variation in alkyl chain length within these *n*OBA molecules influence their flow and deformation behaviour when compared to the *n*BA series. By focusing specifically on homologues with n = 5–8, we aim to uncover correlations between molecular structure and rheological behaviour.

Molecular Structure

Alkoxy- or alkyl-substituted aromatic acids exhibit mesomorphism due to dimer formation through carboxylic acid hydrogen bonding [21]. *p*-*n*-Alkyl(oxy)benzoic acid mesogens are the organic compounds frequently used in the liquid crystal research. Molecules of *n*BA have alkyl groups attached to the benzene ring, while *n*OBA have alkyloxy groups attached, where the alkyloxy groups consist of an alkyl chain linked to the benzene ring through an oxygen atom. Both alkyl(oxy)benzoic acids exhibit a roughly planar molecular shape due to the presence of a benzene ring. However, the attachment of the alkyl group in alkyl benzoic acids and the alkyloxy group in alkyloxy benzoic acids results in slight differences in molecular conformation.

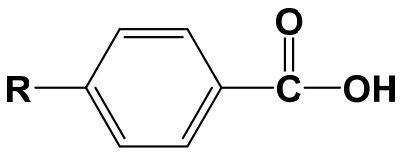


Fig. 1. Molecular structure of mesogenic alkyl(oxy)-benzoic acids, where R= C_nH_{2n+1} (alkyl) and R=OC_nH_{2n+1} (alkyloxy), n – indicates the homologue number

In alkyloxybenzoic acids, carboxylic acid functional groups (HO-C=O) are crucial in the formation of H-bonds. These bonds occur in the region associated with carbonyl (C=O) stretching vibrations and can bond with either the hydroxyl group (–OH) of nearby molecules or with hydrogen atoms attached to the aromatic ring [22]. Because alkoxy benzoic acid molecules contain ether oxygen atoms, they can interact more strongly between layers formed by benzoic acid dimers. This stronger interaction is due to resonance-assisted hydrogen bonding in the *n*OBA series compared to the *n*BA series. Additionally, the highly electronegative oxygen atom contributes to the overall conjugation system within the hydrogen-bonded dimers, further strengthening the hydrogen bonds.

Experimental methods

As cross-polarized visual observation is a faster method for determining and evaluating the boundaries of phase regions, we measured the phase transition temperatures with a Meopta polarizing optical microscope (POM). For rheological measurements, we used a controlled stress rheometer (ARG2, TA Instruments) with a 40 mm parallel plate setup. We heated the samples to their clearing point and isothermally held for 5 minutes. Throughout various temperatures and shear rates, we observed no edge fracture. Since we employed the same experimental methods for both *p-n*-alkyl(oxy)benzoic acid mesogens, we were able to directly compare their specific behaviour under identical conditions. This enabled us to assess the rheological properties of each series without introducing confounding variables from different methodologies.

Results and discussions

Phase behaviour

The studied *n*BA molecules can form a nematic phase. Table 1 displays the phase transition temperatures of *n*BA, which are consistent with values reported in existing literature [23, 24].

Table 1. **Phase transition temperatures of *p-n*-alkyl-benzoic acids**

Mesogens	Mesomorphic range (°C)
5BA	82.0—N—117
6BA	95.2—N—112.5
7BA	102.5—N—117.5
8BA	99.1—N—109.6

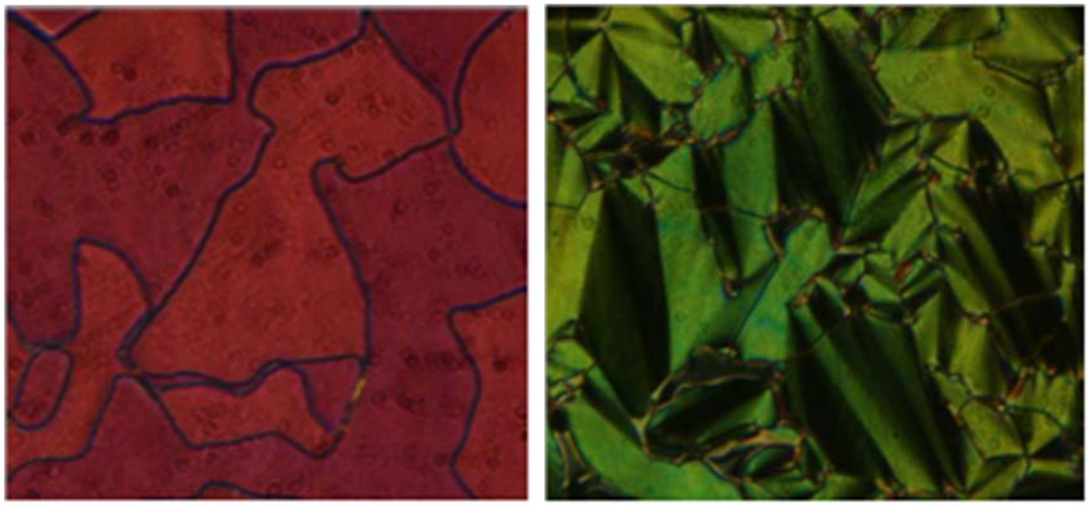
The *n*OBA compounds with n = 5, 6 display schlieren nematic texture, while those with n = 7, 8 exhibit schlieren smectic C texture at lower temperatures [25–28]. It is observed that the temperature ranges over which the liquid crystal exhibits the smectic phase increased with increasing alkyloxy tail length in benzoic acid derivatives. Conversely, the nematic phase temperature range shrinks with longer alkyloxy tails. Table 2 displays the obtained phase transition temperatures. Figure 2 illustrates the nematic and smectic C textures of *n*OBA compounds.

Table 2. **Phase transition temperatures of *p-n*-alkyloxybenzoic acids**

Mesogens	Mesomorphic range (ºC)
5OBA	121.6—N—145.5
6OBA	101.9—N—150.5
7OBA	89.8——SmC———97.5—N—109.6
8OBA	95.1——SmC ——102.4—N—136.2

It has been noted that both the series have showed even-odd effect [29] and *n*OBA series possess higher transition temperatures than *n*BA. This difference can be attributed to the presence of an oxygen atom which introduces electronic effects in the alkyloxy group. This can also be attributed to the *n*OBA series' extended conjugation compared to the *n*BA series, where the hydrogen bond strength is significantly increased due to the involvement of a conjugated π-electron system.

Further, it is noteworthy that the 7BA and 8BA homologues do not exhibit smectic phase, in contrast to representatives of the *n*OBA homologues. The combined effects of disrupted π-π interactions, limited conjugation region and reduced stacking tendency collectively explain the absence of smectic phases in the higher homologues within the *n*BA series [30]. Blending of alkyl(oxy)benzoic acids results in the formation of unique, asymmetric heterodimers. These heterodimers, like their individual homodimer counterparts, can form a mesophase, but with distinct phase transition temperatures and altered viscosity properties. Yadykova et al. demonstrated that equimolar blending of these easily accessible mesogens achieves the liquid crystal phase at lower temperatures [31].



Nematic phase Smectic C phase

Fig. 2. Textures of *n*OBA compounds

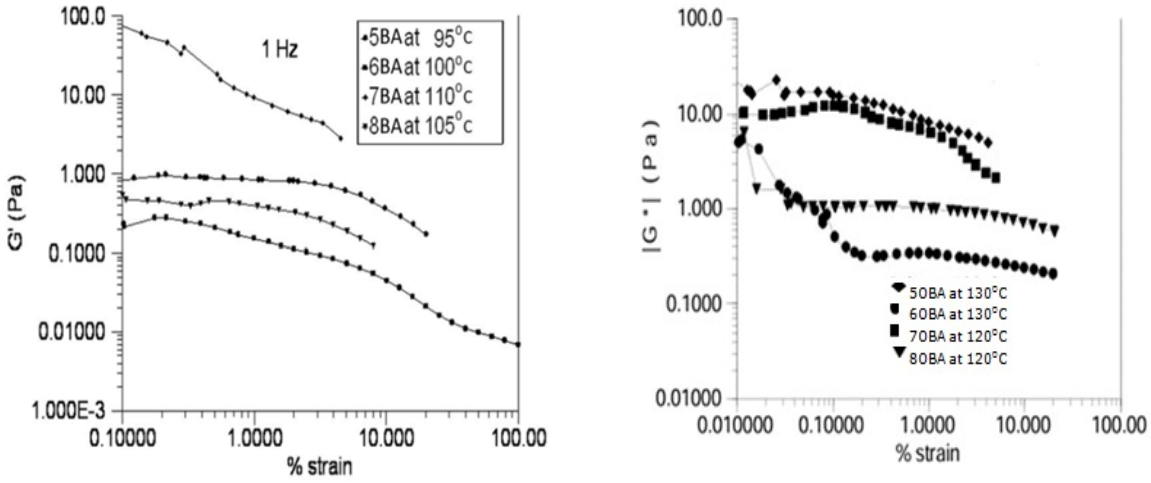
Strain sweep behaviour

In the linear regime of deformation, rheological measurements provide a clear picture of a material's molecular structure because these measurements are more sensitive to molecular interactions compared to the non-linear regime. The strain sweep test gives the specific findings related to the occurrence of nonlinearity and the extent of linear viscoelastic behaviour (LVR) exhibited by the samples. The investigation of LVR is carried out at different frequencies, but focuses on the LVR at 1Hz for both and presented in Fig. 3. The stress is consistent with strain and at low strain amplitudes in both the groups. *n*BA and *n*OBA compounds were tested in their nematic region.

The onset of nonlinear behaviour (the limit strain amplitude – γc) of *n*BA appears independent of the carbon chain length of the molecules. The graph suggests that for 7BA, the applied strain amplitude has exceeded the limit of its linear viscoelastic behaviour. Interestingly, under conditions of high strain amplitudes and frequencies, 5BA, 6BA, and 8BA show a constant value of the linear viscoelastic region.

Contrary to this, the strain at which nonlinear behaviour starts (γc) depends on the carbon chain length for *n*OBA. Even-chain samples show a larger linear region. Odd-chain samples have a lower critical strain, with 7OBA showing nonlinearity at only 0.1 %. It is also observed that 6OBA has the lowest value of complex modulus.

Upon comparison, the extent of LVR is almost the same for both *n*BA and *n*OBA. But the onset of nonlinear behaviour of *n*BA homologues is chain length independent and odd-even effect is observed for *n*OBA homologues.



(*a*) (*b*)

Fig. 3. Strain sweep behaviour of *n*BA (*a*) and *n*OBA (*b*) homologues

Frequency sweep behaviour

In this experiment, the angular frequency is a measure of how quickly the shear stress is applied to the liquid crystal samples. Measuring the storage (G') and loss moduli (G") in a frequency sweep test yields important insights into the viscoelastic behaviour of materials. Figure 4 represents the findings revealed by these measurements. The frequency sweep measurements for each individual compound were conducted within its own linear viscoelastic region (LVR).

The influence of chain length and frequency on the viscoelastic behaviour of *n*BA mesogens demonstrated a transition from liquid-like to solid-like response at high frequencies and stronger elastic character with longer chains. However, the specific frequency dependence of G' and G" varied with chain length. For homologues with n = 5–7, the storage modulus (G') consistently exceeded the loss modulus (G") at the highest frequencies tested. In contrast, for 8BA, the loss modulus (G") was observed to be higher than the storage modulus (G'). The variation of moduli with chain number of *n*BA homologues is presented in Fig. 4. Crossover frequencies and relaxation times are identified for each compound. For 5BA and 6BA the viscous character has dominated and in the case of 7BA and 8BA, the storage of elastic energy has prevailed over the dissipation of viscous energy. These results are consistent with viscosity measurement in steady shear experiment showing higher viscosity.

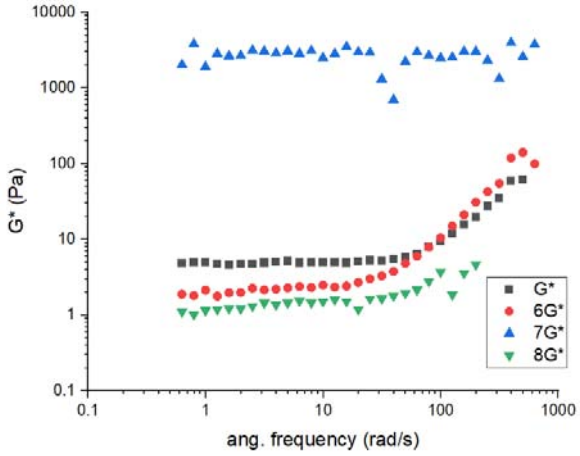


Fig. 4. Variation of complex modulus (G*) of *n*BA homologues

In the *n*OBA series, it is common for smectic phases to display a higher complex modulus when compared to nematics. It is noticed that storage and loss moduli of 6OBA are lesser than other homologues. Based on a previous study on texture transitions 6OBA, this material marks the first instance of a texture transition occurring directly from a crystalline phase to a nematic phase [32]. These sub phases could potentially decrease viscoelasticity by inducing disorder or disrupting the molecular arrangement of the liquid crystal.

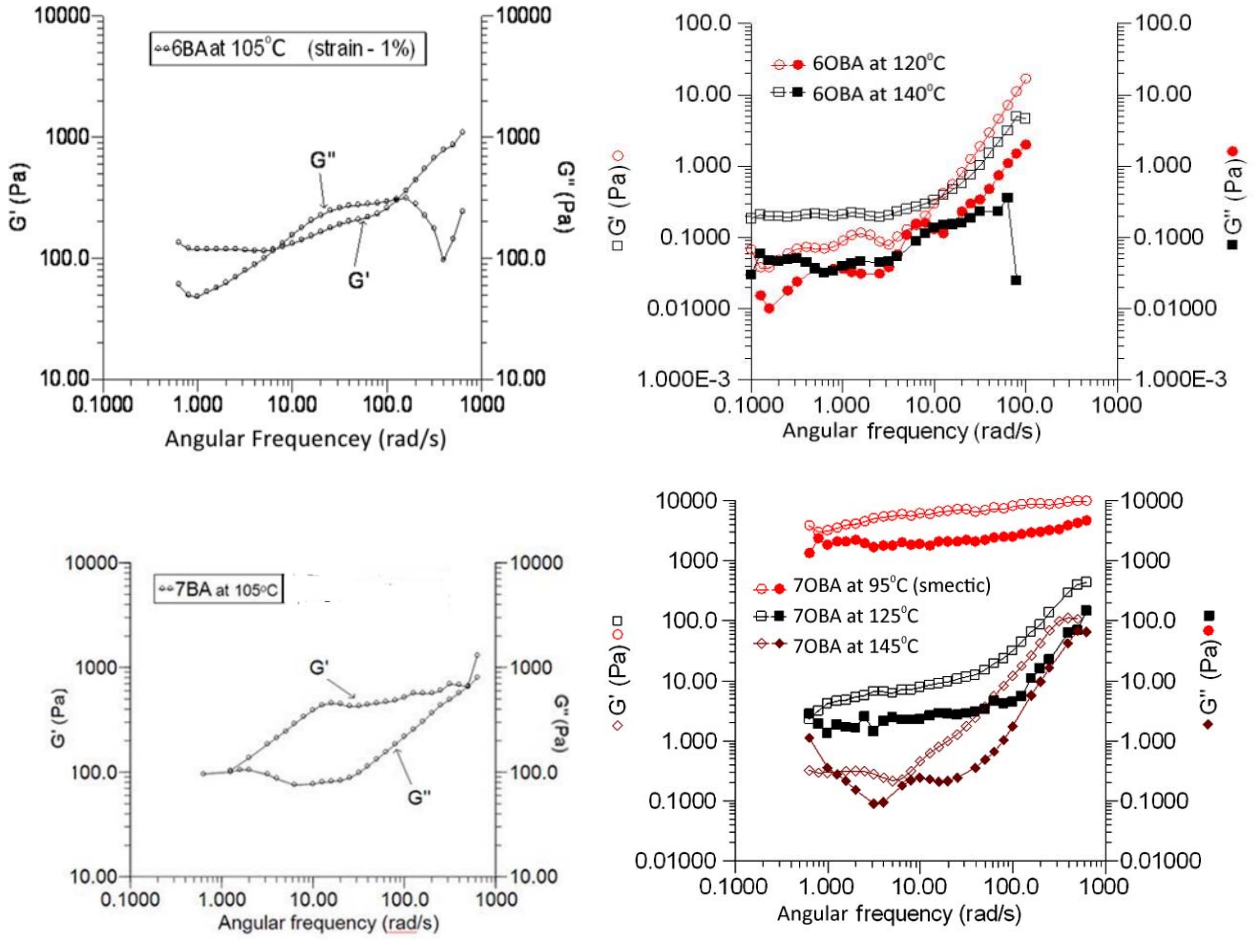


*Fig. 5.*Variations in the storage modulus (G′) and the viscous modulus (G″) with frequency

Upon comparison, at low frequencies, the homologues of both series exhibit similar behaviour, displaying a frequency-independent plateau in the moduli. This suggests that the material contains cooperative rearrangements as in rubber polymers [33]. Figure 5 illustrates variations in the storage modulus G′ and the viscous modulus G″ with frequency for nematic and smectic phases. However, the storage and loss moduli of *n*OBA homologues are lower than those of *n*BA homologues. The storage modulus or loss modulus is primarily determined by intermolecular interactions, molecular packing, molecular mobility, and aspect ratio, which may differ between alkyl(oxy)benzoic acids regardless of their transition temperatures.

Steady shear behaviour

The steady shear test has a substantial impact on liquid crystal rheology. It provides useful information regarding a liquid crystal's flow behaviour, elasticity, and appropriateness for different technological applications.

The *n*BA homologues described the shear thinning behaviour at low shear rates, characterized by a decrease in viscosity with increasing shear rate. Which is a distinct feature compared to isotropic liquids. This is attributed to the alignment of rod-like molecules in the flow direction under shear, reducing resistance to flow. The viscosities exhibited a three-region flow curve, consistent with the Onogi-Asada model.

At low shear rates, the viscosity displayed a slight dependence on molecular structure. However, at higher shear rates, the deformation of the molecular chains dominated the behaviour, leading to a decrease in viscosity (Region II). Ultimately, a constant viscosity was reached (Region III). This pattern was consistently observed across the studied materials. Region I would typically show a constant viscosity. It is observed that at low shear rates, 6BA has higher viscosity and 5BA has least viscosity over the range of shear rates. While investigating the physical, chemical, optical and insulating properties of alkyl benzoic acids, Mirtunjai Mishra et al., observed that 5BA has the highest O–H bond stretching [34]. The stretching of O–H bond can induce a decrease in viscosity with increase in shear rate. If 5BA has O–H bonds that can stretch significantly, it means these bonds are quite weak and flexible. This flexibility could make it easier for molecules to line up, causing the liquid to flow more easily, which means that it would have low viscosity. Higher shear forces and lower viscosity ratios are commonly noted in liquid crystals experiencing substantial molecular realignment under shear stress. Among the members of the series under our study, pentyl benzoic acid (5BA) exhibits a high value in a specific ratio, suggesting a strong alignment of its molecules.

For *n*OBA, steady shear behaviour aligns with the Carreau model [35], featuring a power index ranging from 0.7 to 1.2. In the smectic phase, the viscosity of 8OBA at low shear rates resembles that of robust gels. In the nematic phase, there is shear thinning at low strains, followed by a consistent viscosity plateau at high strains. On the other hand, the smectic phase displays no plateau and undergoes continuous variation with strain. We observe that the smectic phase of *n*OBA exhibits non-Newtonian viscosity, with the highest viscosity observed for 7OBA. Kucherepa et al. [36] observed similar results and attributed them to strong interlayer interactions. These interactions arise from dimers formed by hydrogen bonds between carboxylic acid groups and ether oxygen atoms, along with aromatic protons. As the length of the aliphatic chain increases, the molecules gain greater mobility, weakening the influence of hydrogen bonding relative to the weaker van der Waals forces. Hence the viscosity of 8OBA consequently falls in comparison to the 7OBA homologue. However, at temperatures corresponding to the nematic phase and particularly the isotropic liquid phase, Newtonian viscosity was observed. The smectic phase of 7OBA is much higher than other homologues including smectic phase of 8OBA due to odd-even effect. At high shear rates, molecules may not always perfectly align and can undergo a tumbling motion that partially aligns and then rotates; the extent of shear thinning is influenced by the molecular structure and rigidity of the molecules in the liquid crystal. Here also, there is evidence to suggest that 6OBA has lowest shear viscosity. It is also mentioned that 6OBA has oligomers [29] near the clearing point. Considering this, we assume that these oligomers are not well-aligned or they disrupt the ordering of molecules, contributing less to viscoelasticity and viscosity compared to other homologues.

In the context of steady shear, Fig. 6 illustrates that both alkyl(oxy)benzoic acids share similarities such as shear thinning. Notably, the viscosity of *n*OBA homologues is observed to be lower than that of their *n*BA counterparts. The shear viscosity of a substance depends on several factors, such as its molecular structure, intermolecular interactions, and arrangement of molecules. The discrepancies in molecular structure can cause differences in shear viscosity. According to the published crystallographic data [30], every molecule within the *n*BA series is distinguished by a non-planar configuration and *n*OBA with planar structure. Non planar structure results in complicated molecular arrangements and interactions, which make it harder for them to flow smoothly, like traffic jams on a busy road. Whereas planar molecules of *n*OBA generally have more uniform molecular arrangements and allow for more efficient molecular alignment under shear stress, leading to smoother flow and potentially lower shear viscosity. Mixing these liquid crystals in equal parts creates a new mixture with a lower viscosity than either of the original liquid crystals by themselves [31].

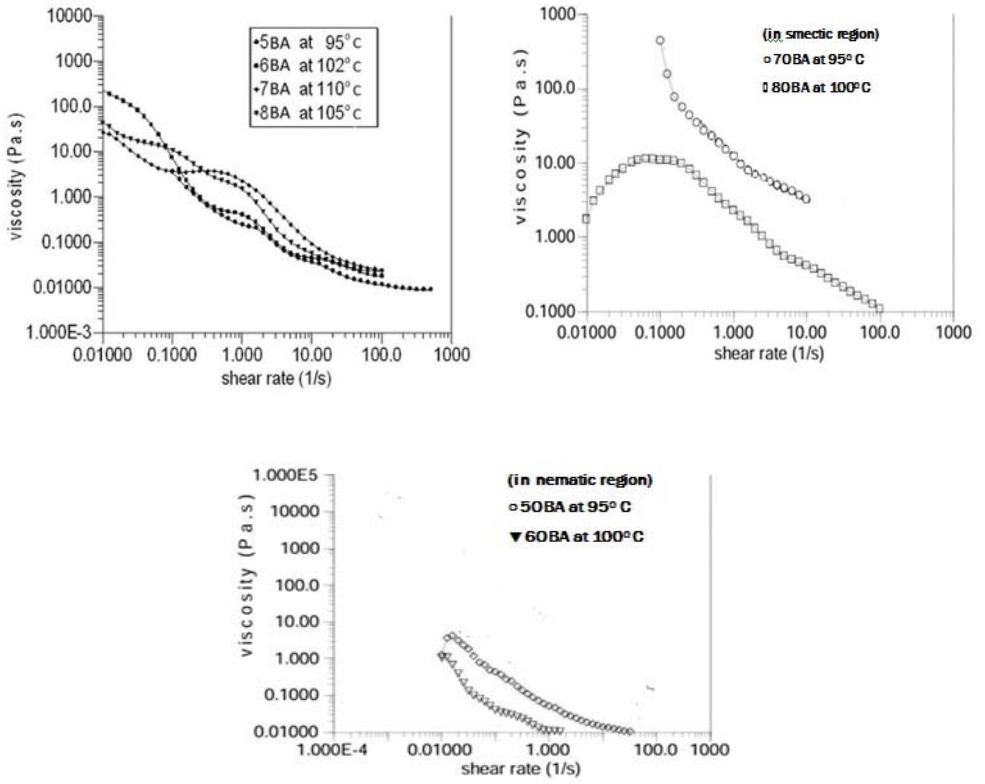


Fig. 6. Shear rate vs viscosity for alkyl(oxy)benzoic acids in the nematic and smectic regions

Our studies on flow properties reveal some key differences between the *n*BA and *n*OBA series. The presence of oxygen atoms in the *n*BA series seems to lower the temperature at which they undergo a phase transition (change in molecular arrangement). The non-planar structure of *n*BA molecules further hinders their flow compared to the flatter *n*OBA molecules. Overall, both *n*BA and *n*OBA series exhibit interesting flow properties making them appropriate for a range of uses based on particular requirements. The *n*BA series is highly suitable for use in absorbance or transmittance application devices. Additionally, at room temperature, these molecules exhibit excellent performance in insulating application devices [31].

The rheological properties of alkyloxybenzoic acids play a vital role in determining their effectiveness across various industrial applications, especially in lubrication. Benzoic acids are extensively utilized as additives in cutting fluids and lubricants, to improve performance. According to a study on alkyloxy benzoic acid antimicrobial activities, 4OBA, 6OBA, and 7OBA demonstrated a passable level of antibacterial activity in a spent coolant. Additionally, in a test using cast iron chips, aqueous solutions of 6OBA demonstrated outstanding corrosion resistance [37].

Research has shown that not only do pure benzoic acids exhibit good lubricating properties, but their combinations such as the 4-acryloyloxyphenyl ester of 4'-*n*-8OBA and a system consisting of 3OBA and *p*-*n*-propyloxy-*p*'-cyanobiphenyl in liquid paraffin, were examined under boundary friction conditions These studies, conducted on both pure substances and their solutions in a liquid paraffin base, revealed that the tribological properties are influenced by the chemical structure and concentration of the additives. The presence of these triboactive additives in lubricants not only enhances lubrication performance but also stabilizes it under heating highlighting the versatility

and potential of benzoic acid-based compounds [38]. The cholesteryl ester of *p*-*n*-dodecyloxybenzoic acid exhibits a broad temperature range for its mesomorphic phase, spanning from 127 to 195 °Cand demonstrated best lubricating properties. In the tribosystem, it also shows the lowest friction coefficient ranging from 0.06 to 0.08 [39]. Two derivatives of 4-dodecyloxybenzoic acid [40] were evaluated as antioxidants for Egyptian base oil. The compounds were evaluated by monitoring changes in both total acid number and viscosity.

Conclusion

The oscillatory viscoelastic properties and steady shear viscosities of *p*-*n*-alkyl(oxy)benzoic acids are compared and explained. The extent of linear viscoelastic region is almost the same for both *n*BA and *n*OBA. During frequency sweep, at low frequencies, the homologues of both series exhibit similar behaviour, displaying a frequency-independent plateau in the moduli. The steady shear behaviour of *n*BA aligns well with Onogi-Asada's three region model and of *n*OBA with Carreau model. The viscosity of *n*OBA homologues is observed to be lower than that of their *n*BA counterparts. The inclusion of oxygen in alkyloxy benzoic acids modifies their molecular interactions, resulting in increased transition temperatures and low viscoelastic properties in comparison with alkyl benzoic acids. These findings offer valuable insights into potential applications across various fields like lubrication applications.

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