



## Test Method

# Measurement of adsorption energy between a solid adsorbent and a liquid adsorbate using differential scanning calorimetry



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## ABSTRACT

We report on a novel method involving the use of differential scanning calorimetry (DSC) in evaluation of adsorption energy between a liquid adsorbate and a solid adsorbent. The proof of concept is demonstrated by measuring the exothermic heat release due to the adsorption of automotive transmission fluid (ATF), the adsorbate, to a paper-based friction material used in automotive torque converters, the adsorbent. The novelty of the measurement technique involves initial freezing of the liquid adsorbate so that the initiation of the adsorption process can be identified. Our experimental results and theoretical calculations reveal that the adsorption energy of the friction paper and the summation of adsorption energies of each friction paper ingredient are in good agreement.

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## 1. Introduction

Friction materials have long been used in automotive industry together with automotive transmission fluid (ATF). However, the complexity of the interactions between each of the components led to developments that are based on a trial-and-error method [1].

ATF is a special fluid that consists of approximately 80% of a type of base oil. The remaining 20% of the fluid consists of viscosity modifiers, pour point depressants, foam inhibitors and performance additives which are mainly friction modifiers, detergents, dispersants, etc. Performance additives are amphiphilic molecules in which nonpolar tails provide oil solubility and polar heads provide interaction with the surfaces due to their high activity. Another advantage of performance additives is the alignment of hydrocarbon tails perpendicular to the sliding surfaces during boundary lubrication regime which provides effective lubrication.

Previously, it was found that adsorption of ATF additives on friction material and steel surfaces in a torque converter clutch (TCC) improves the performance of an automatic transmission while preventing shudder [2]. The desired friction versus velocity ( $\mu$ - $v$ ) curve with a positive slope for a TCC can be obtained by creating a well-developed monolayer film of friction modifiers on the surfaces. It is provided by the smooth transition from static to

dynamic conditions. Therefore, the adsorption of ATF additives on the sliding surfaces of a clutch plate is very important [3]. These additives prevent the direct contact of the asperities during the boundary lubrication regime even after the depletion of the lubricant molecules due to various clutch engagement conditions.

Adsorption can be classified into two groups as physisorption in which the energy release is relatively low due to van der Waals interactions and chemisorption in which higher energy is released due to formation of chemical bonds. Because of the presence of so many different chemical functionalities together with possible interaction of these functionalities with externally applied physical parameters such as temperature, pressure, and speed, it is difficult to predict the exact adsorption mechanism between ATF and the surfaces. Friction modifiers with carboxylic acid functionality can be chemisorbed on to metal surfaces; whereas, a friction material surface with hydroxyl and siloxane functionality will more likely involve physisorption [4,5].

Adsorption studies of molecules similar to ATF performance additives and possible friction material components have been performed by various researchers using different techniques. Ellipsometry [6], quartz crystal microbalance (QCM) study of fatty acid adsorption from alkane solution [7], scanning tunneling microscope (STM) imaging of fatty acids on graphite [8], titration measurements of stearic acid onto kaolinite [9], or Celtek clay [10], adsorption energies of ATF additives onto various friction material ingredients by flow micro calorimetry measurements [2] are some of the techniques employed previously. Among these techniques

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only the work done by Kamada et al. quantifies the adsorption energies between wet friction materials and ATF ingredients which allows comparison. However, the report of 0 J/g energy between some ingredients arises some questions [2]. Although these studies provided some information about adsorption properties, the techniques employed are too simplified and of limited capabilities, and thus, not suitable to provide an insight to the complex medium of a wet friction materials.

In our studies, differential scanning calorimetry was used to measure the exothermic heat release due to the adsorption of an ATF, the adsorbate, to a wet friction paper used in torque converters, the adsorbent. A commercial ATF was procured to be used for the adsorption measurements. The ATF used in the experiments contained over 85% base oil. It also contained a methacrylate polymer which is a viscosity modifier, succinimides which are dispersants, and dialkyldithiophosphates which are antiwear agents, in percentages presented in Fig. 1. First, theoretical estimates on the limits of total adsorption energies possible were calculated considering the maximum and minimum amounts of additives in ATF. For these calculations, average molecular weight values were used for the specific molecules involved and listed above [11]. The adsorption of base oil was assumed to be due to dipole-induced dipole interactions (2 kJ/mol) and adsorption of ATF additives was assumed to be due to either dipole-dipole interactions or hydrogen bonding (8–42 kJ/mol) [12]. Based on these calculations and for cases in which the amount of ATF would be the limiting factor in the adsorption process, depending on the amounts of the adsorbate and the adsorbent involved, the energy measurements were expected to be approximately between 4 and 7 kJ/g, as shown in Fig. 1.

## 2. Experimental

### 2.1. Materials

A paper-based friction material (friction paper) consisting of synthetic fibers, organic fibers, cured resin, and diatomaceous earth were provided by LuK LLC USA (Wooster, OH) in both composite and individual component forms. The wet friction papers were

tested in two different forms, as solid discs with 3 mm diameter, and also in pulverized form. The cured resin was ground into micron size particles using a blender while all the other adsorbents were used as received, in their natural state.

In addition to the commercial ATF that was used as an adsorbate, a group III base oil and base oil with friction modifier having different concentrations were provided by The Lubrizol Corporation (Wickliffe, OH). 2 wt % stearic acid was added into the commercial ATF for limited number of adsorption measurements.

### 2.2. Characterization techniques

Thermogravimetric analysis (TGA) (TA-Instrument Q50) was used to determine the operational temperatures of each component and to choose the temperature range to be used in differential scanning calorimetry.

A TA-Instrument Q2000 with an attached liquid nitrogen system which allows cooling down to  $-180\text{ }^{\circ}\text{C}$  was used for differential scanning calorimetry (DSC).

### 2.3. Procedure

It is not possible to measure the adsorption energy between a liquid adsorbate (ATF) and a solid adsorbent (wet friction paper or its ingredients) using DSC at room temperature because the adsorption occurs as soon as the materials are brought into contact with each other, with adsorption occurring spontaneously and immediately. Therefore, by the time the DSC instrument starts the measurement, the adsorption had already occurred. In order to eliminate this problem, the ATF was frozen separately from the adsorbents before bringing the adsorbent into contact with ATF at  $-140\text{ }^{\circ}\text{C}$  in the DSC pan. The adsorbate was placed into the DSC sample pan while the adsorbent (friction paper) was placed into the lid which was set upside-down. The sample pan was placed onto the sample platform and the lid was placed on top of the reference pan and lid (Fig. 2). The temperature of the sample was increased to  $120\text{ }^{\circ}\text{C}$  followed by 10 min of isothermal heating to remove the moisture. Then, the sample was cooled down to  $-140\text{ }^{\circ}\text{C}$  and the

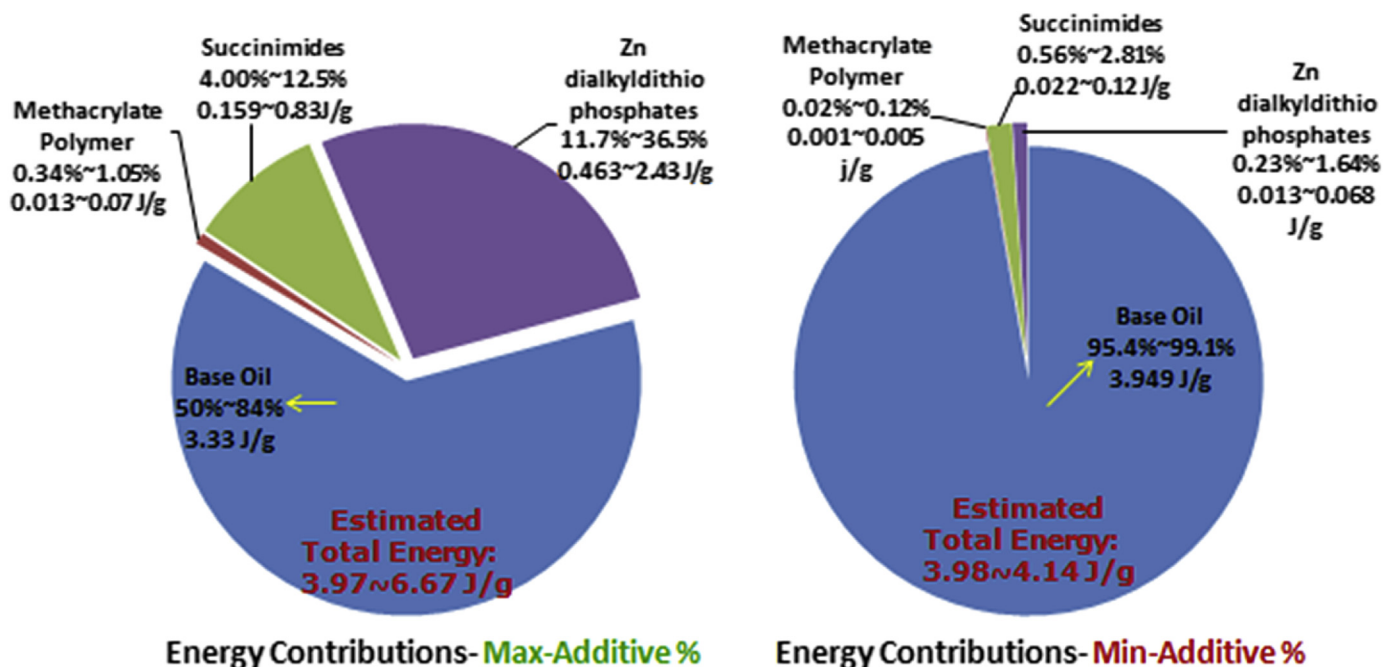


Fig. 1. Adsorption energy contributions of ATF components based on theoretical calculations.

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