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Adsorption and adhesion energies of *n*-Decane on the Pt(111) surface by calorimetry

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ABSTRACT

The heat of adsorption of *n*-decane molecularly adsorbed on Pt(111) at 150 K was measured by single-crystal adsorption calorimetry (SCAC). The heat of adsorption was initially ~152 kJ/mol on terrace sites up to a coverage of $\Theta = 0.045$ ML . Previous TPD measurements of the desorption energies of shorter n-alkanes (C_1 to C_6) on Pt(111) terraces showed a linear increase with chain length which predicts a desorption energy for n-decane in very good agreement with this new result. A heat of 169 kJ/mol was measured below 0.004 ML and attributed to defect sites, probably step edges. At coverages above $\Theta = 0.27$ ML (~3 molecular layers), the multilayer heat of adsorption was constant at ~85 kJ/mol. Using the heat of adsorption versus coverage at 150 K up through this bulk-like multilayer, the adhesion energy per unit area of liquid *n*-decane on Pt(111) terraces at room temperature was estimated to be 0.148 J/m^2. This is very similar to the value estimated for liquid *n*-hexane on Pt(111) from TPD energies, which shows that while adsorption energy increases nearly proportional to the number of CH_2 plus CH_3 groups, as the adhesion energy is a per unit area value, it remains nearly constant (0.15 J/m^2) between hexane and decane.

1. Introduction

The choice of solvent is known to have a dramatic effect on solid-catalyzed reaction rates and selectivities,[1–8] and understanding these effects is important for tuning catalyst performance. It is of great interest to understand the energetics of solvent bonding to catalytically relevant materials, since these energetics to a large extent determine how different solvents affect the heats of adsorption of catalytic reaction intermediates to the catalyst surface, and their differences with respect to heats of adsorption in the gas phase (which are much easier to measure and much easier to computationally estimate, and thus are much more widely known). Specifically, it has been shown theoretically that the heat of adsorption of neutral reaction intermediates decreases in a particular solvent (relative to the gas phase) by an amount approximately equal to the adhesion energy per unit area (E_{adh}) of that liquid solvent to the surface times the area on the surface occupied by that adsorbed intermediate (i.e., the area where solvent can no longer bind to the surface due to blocking by the adsorbate).[9–12] Since the energies of adsorbed intermediates and transition states determine catalytic reaction rates, understanding how different solvents affect adsorption energies of intermediates aids in our fundamental understanding of solvent effects on catalytic activity and selectivity, and in the improvement and design of catalytic processes. Here we report calorimetric measurements of adsorption energy versus coverage for *n*-decane on Pt(111) at 150 K, and use these gas phase measurements to estimate the adhesion energy of liquid *n*-decane on Pt(111) at room temperature. We compare this adhesion energy to the estimated energies of other linear alkanes on the Pt(111) surface to examine the relationships between adsorption energy, adhesion energy and size of these linear alkanes. The adsorption energy is found to be nearly proportional to chain length, whereas E_{adh} is nearly independent of chain length.

The adsorption energy of *n*-decane on Pt(111) is also of fundamental interest since hydrocarbon conversion reactions on Pt catalysts are ubiquitous in petroleum and biomass catalysis, decane is a common component in these reaction mixtures, and the Pt(111) surface is the most widely studied model of Pt catalyst surfaces. Due to the growing need to convert polymer waste to valuable products, the reactions of very long alkanes (polyolefins) on Pt and similar metal catalysts has recently become a subject of great interest. Thus, it is important to understand the relationship between alkane chain length and adsorption strength of linear alkane systems. Neither the adsorption energy nor desorption activation energy of *n*-decane were previously known. While the desorption energies of shorter linear alkanes (C_6 and smaller) on Pt(111) were previously measured using temperature programmed desorption (TPD), *n*-octane and *n*-decane did not desorb completely reversibly (they partially dissociated instead), and thus their desorption

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energies could not be measured using TPD.[13] As for other alkanes on Pt(111), [14,15] this dissociation occurs in competition with desorption during TPD near the desorption peak temperature. Single crystal adsorption calorimetry (SCAC) allows for the direct measurement of the heat of adsorption versus coverage of molecules that do not desorb reversibly.[16] This paper thus reports the heat of adsorption versus coverage of *n*-decane, the largest alkane that has been studied on Pt (111). We study it here at 150 K, well below the desorption temperature of *n*-hexane in those TPD studies (225 K) [13], so that negligible dissociation is expected to occur. With a small correction, this heat of adsorption can be converted to the desorption activation energy like that measured by TPD.[17]

2. Experimental

The experiments were conducted in an ultrahigh vacuum (UHV) chamber (base pressure $<2 \times 10^{-10}$ mbar) designed for single-crystal adsorption calorimetry (SCAC). The chamber also has in-situ instrumentation for X-ray photoelectron spectroscopy (XPS), low-energy electron diffraction (LEED), Auger electron spectroscopy (AES), low-energy ion-scattering spectroscopy (LEIS), a quadrupole mass spectrometer (QMS), and a liquid-nitrogen cooled quartz crystal microbalance (QCM). The SCAC apparatus and experimental procedures for the molecular beam flux, sticking probability, and heat measurements have been described in detail previously. [18–20]

In brief, the 1μm thick Pt(111) single crystal sample was purchased from Bine Hansen at Aarhus University. The crystal surface was cleaned by repeated cycles of Ar^+ ion sputtering and annealing to 1120 K, which was proven in earlier studies with this same apparatus to give a clean and well-ordered Pt(111) surface.[21] The Pt(111) surface was exposed to a pulsed, collimated molecular beam of *n*-decane, and the heat of adsorption and sticking probability were recorded simultaneously. The sticking probability was measured by the King and Wells method with a QMS, and the heat of adsorption was measured using a pyroelectric polymer ribbon in contact with the back of the Pt(111) crystal. The molecular beam was created by expanding 1.0 mbar of *n*-decane through a glass capillary array heated to 335 K and collimated via a series of liquid-nitrogen cooled orifices. This beam was then chopped into 102 ms pulses every 3 s. During the course of these measurements, there was an extra contribution to the heat signal's noise that was only discovered after the experiments reported here, so the signal-to-noise ratio on heats reported here is significantly lower than is typical for this calorimeter.

Here, one monolayer (ML) of coverage is defined as the number of molecules of *n*-decane adsorbed to the surface per unit area, normalized to the surface density of Pt(111) atoms (1.50×10^{19} molecules/m^2). A typical beam pulse consists of approximately 0.002 ML ($\sim 4.7 \times 10^{11}$ molecules within the beam diameter of ~4 mm) per *n*-decane gas pulse.

3. Results

3.1. Sticking probabilities

Sticking probabilities were measured by comparing the time-integrated QMS signal for *n*-decane when the molecular beam scatters from the Pt(111) surface to that from a gold surface (after a saturation dose), which is known to have zero sticking probability for *n*-decane. When the signals are the same, the sticking probability is zero. Long-term and short-term sticking probabilities of *n*-decane on Pt(111) were found to be unity at 150 K. The long-term sticking probability refers to molecules that initially adsorb and remain adsorbed for the entire duration of the 3 s pulse window and contribute to the build-up of coverage of *n*-decane on the surface. The short-term sticking probability refers to the molecules that initially adsorb and contribute to the measured heat of adsorption during the 102 ms pulse, but then might desorb again before the next pulse and not add to the coverage. When both sticking probabilities are unity, as with *n*-decane on Pt(111) at 150 K here, all incoming molecules adsorb to the surface and remain adsorbed until the next pulse, fully contributing to the both the measured heat and coverage increase.

3.2. Heat of adsorption

We define here the "heat of adsorption" as the negative of the standard molar enthalpy change for the adsorption reaction, with the gaseous molecules and single crystal surface being the same temperature. As explained in detail in previous papers, this requires a small enthalpy correction on the measured heat as the gas molecule's enthalpy at this temperature is slightly different than a Boltzmann distribution at the sample temperature due to the actual molecular beam conditions.[20] The differential heat of adsorption measured is the heat released per mole when a pulse from the molecular beam is dosed onto the crystal surface at nearly fixed coverage, as is the case during our SCAC experiments here since the gas pulses contain only ~0.2 % of a ML. Fig. 1 shows the differential heat of adsorption of *n*-decane on Pt(111) at 150 K as a function of *n*-decane coverage. This is the average of 6 experimental runs. After the first two gas pulses, the heat of adsorption at 150 K is nearly constant at 151.8 ± 5.4 kJ/mol up to 0.045 ML. The heat is slightly higher in the first two points (~169 kJ/mol), which has often been seen in SCAC measurements on the Pt(111) surfaces previously studied in this apparatus. This has been attributed to defect sites on the surface, likely step edges. [22] Above 0.06 ML, the heat of adsorption drops rapidly to ~90 kJ/mol at ~0.09 ML. It stays near 90 kJ/mol until above ~0.20 ML, then decreases to a constant multilayer heat of 84.9 kJ/mol above 0.27 ML which corresponds to coverage of more than three layers of *n*-decane on the Pt(111) surface.[23] The run-to-run standard deviation on this multilayer heat was 5.5 kJ/mol. Since there were six runs, the error bar on the *average* multilayer heat is smaller ($\sim \pm (5.5 \text{ kJ/mol})/(6\text{-}1)^{1/2} = \pm 2.5$ kJ/mol). This average multilayer heat is 2.5 kJ/mol below (but within this error bar of) the heat of sublimation of bulk n-decane(solid) at this temperature, which we estimate to be 87.4 kJ/mol at 150 K based on literature values for the heat capacities of solid, liquid and gaseous *n*-decane, and the enthalpies of vaporization and fusion[24,25].

Linear n-alkanes on Pt(111) have first-layer packing densities that depend on the number of carbons per alkane, n, whereby $1/(n+1)$ is the coverage (per Pt surface atom) of the first layer of adsorbate.[23] For *n*-decane, where $n = 10$, this predicts a coverage of $1/11 = 0.091$ ML for completion of the first layer. This is in excellent agreement with the abrupt change in the slope of heat versus coverage, where the high heat measured in the first layer has deceased rapidly to 90 kJ/mol (at

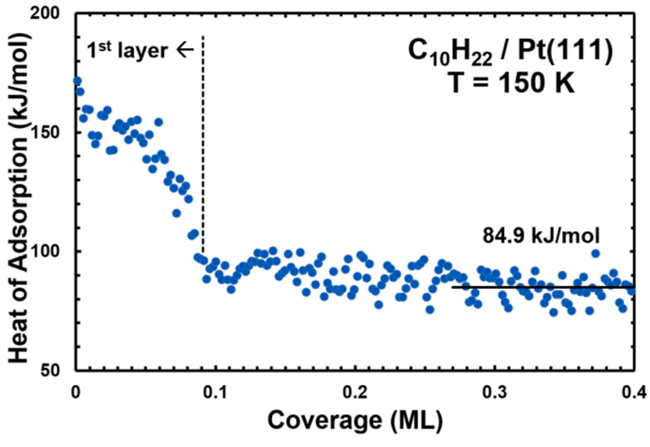

Fig. 1. Differential heat of adsorption of *n*-decane on Pt(111) at 150 K as a function of *n*-decane coverage. The average heat of adsorption reached at the thick multilayer limit is shown by the black line. (1 ML = 1 molecule per Pt surface atom, or 1.50×10^{19} molecules / m^2.

completion of the first layer), and then quickly becomes nearly constant with coverage at ~90 kJ/mol (within the second layer).

4. Discussion

4.1. Comparison to TPD

Previously Tait et.al.[13] measured the desorption energies of various *n*-alkanes on Pt(111), C(0001)/Pt(111) and MgO(100). However, on Pt(111) the longest alkane that reversibly desorbed without significant dissociation was *n*-hexane, so desorption energies for longer alkanes were not studied there. Here, measuring the heat of adsorption of *n*-decane on Pt(111), and correcting for comparison to desorption energy values measured from TPD by subtracting ½ RT,[17] we can compare these experimental results to the value predicted in that work. Taking the linear (near proportional) trend line reported by Tait et al. for the desorption energies for *n*-alkanes versus chain length (from C_1 to C_6) on Pt(111) terraces at a coverage of 0.5 "ML", we predict a desorption energy of *n*-decane on Pt(111) of 129.5 kJ/mol. It is important to note that Tait et al. defined 1 "ML" as their highest measured coverage where they saw only the monolayer desorption peak with no multilayer peak visible, which is close to one complete layer of *n*-alkanes adsorbed on the surface. We define 1 ML here differently, as one molecule of *n*-decane adsorbed to the surface per surface Pt atom (or 1.50×10^{19} molecules/m^2). As Tait et al. measured desorption energies at 0.5 "ML" by their definition, this corresponds to 0.5×0.091 ML $= 0.0455$ ML of decane in our ML units here.

We measured a differential heat of adsorption at 0.0455 ML of 151.8 kJ/mol, which corresponds to a desorption energy of 151.2 kJ/mol after applying the -½ RT correction for direct comparison to TPD measurements.[17] This is 21.7 kJ/mol higher than the desorption energy of 129.5 kJ/mol predicted above at this coverage (i.e., half the coverage which completes the first layer) based on those TPD studies of shorter alkanes by Tait et al.. Those TPD studies indicate that, near the peak temperature for desorption, these alkanes make close-packed 2D islands over nearly the full first-layer coverage range, where their desorption energy remains nearly constant with coverage. Our measured integral heat at completion of the first layer (0.091 ML) is 132.7 kJ/mol, which corresponds to a desorption energy of 132.1 kJ/mol. This average heat of adsorption for the first layer is very close to the average value of 129.5 kJ/mol predicted by TPD measurements of shorter alkanes (see above).

The nearly constant heat of adsorption observed up to 0.045 ML (except at defects) seen in Fig. 1 suggests adsorbed *n*-decane forms 2D islands, which was observed for the other shorter *n*-alkanes studied on Pt(111), and also for linear alkanes up to decane on MgO(100) and graphene, by Tait et al..[13] The decrease in heat seen here above 0.06 ML (~2/3 of the first layer completion) may be due to the lower temperature studied here (150 K, versus 200 and 250 K for MgO and graphene, respectively). Decane molecules on Pt(111) at 150 K here may not be mobile enough at high coverages to achieve their optimum packing configuration to maximize decane-decane attractions. Note too that decane's higher heat of adsorption on Pt(111) suggests that it will have higher surface diffusion barriers than on MgO and graphene, making it even slower to find the minimum-energy packing.

4.2. Adhesion energy

There is great interest in understanding the energetics of solvents at the liquid-solid interface. The adhesion energy at the solvent-metal interface is the controlling factor in the differences between reactant adsorption energies in various solvents relative to its gas-phase adsorption energy.[9–12] Using the method previously described by Rumptz et al.,[11] we can estimate the adhesion energy of bulk liquid *n*-decane to Pt(111) at room temperature from the heats of adsorption in Fig. 1 at 150 K. Eq. 1 shows how the adhesion energy per unit area (E_{adh}) is estimated in that method from the integrated heat of adsorption of the solvent (S), here *n*-decane on the Pt(111) surface:

$$E_{adh} = \frac{\left[Q_{adsorption} - n\Delta H_{vap,S}\right]}{A} + 2\gamma_{S,liq} \tag{1}$$

In the first term, the integrated heat of adsorption at a thick multilayer coverage ($Q_{adsoprtion}$) minus the number of moles adsorbed (n) times the molar heat of vaporization of the liquid solvent ($\Delta H_{vap,S}$) is divided by area covered by the adsorbed film (A). The second term is twice the surface energy of the solvent (decane here) as a bulk liquid ($\gamma_{S,liq}$). For this, we use the reported surface tension of bulk, liquid decane at 298 K, $\gamma_{S,liq} = 0.02383$ J/m^2.[12] It is only possible to form multilayers of *n*-decane on Pt(111) in ultrahigh vacuum at low temperatures (here 150 K), where the incoming molecules adsorb to form a solid instead of a liquid. We make the assumption that $[Q_{adsorption} - n\Delta H_{vap,S}]$ at room temperature is approximately equal to $[Q_{adsorption} - n\Delta H_{sub,S}]$ measured at our lower experimental temperature of Fig. 1 (150 K), where $\Delta H_{sub,S}$ is the heat of sublimation of bulk *n*-decane at this same temperature (taken to equal the limiting multilayer differential heat measured in Fig. 1 at thick multilayer coverages, 84.9 kJ/mol). This assumes that the molar heat capacity of the solvent molecules in the first adsorbed layer equals that in the bulk multilayer at all temperatures between 150 K and 298 K (including the contributions from their heats of fusion). [11,12] In this way, we estimate from the data in Fig. 1 that the adhesion energy of *n*-decane on Pt(111) is 0.148 J/m^2.

In Fig. 2 we plot the adhesion energies of various *n*-alkanes on Pt(111), graphene on Pt(111), and MgO(100) surfaces versus chain length, n. Except for decane on Pt(111), these points are all taken from Rumptz et al.,[12] who extracted them from TPD measurements[13] also using Eq. 1.

Based on the trends seen in Fig. 2 for MgO and graphene and as discussed by Rumptz et al. [12] we expected that the adhesion energy of linear alkanes would be approximately constant as chain length increases. Although the molar adsorption energy of *n*-decane is higher than that of the shorter alkanes (and nearly proportional to chain length), [13] adhesion energy is a per area value (J/m^2), and the adhesion energy does indeed remain the nearly the same for decane as seen for *n*-hexane on Pt(111), since each CH_2 and CH_3 group adds nearly

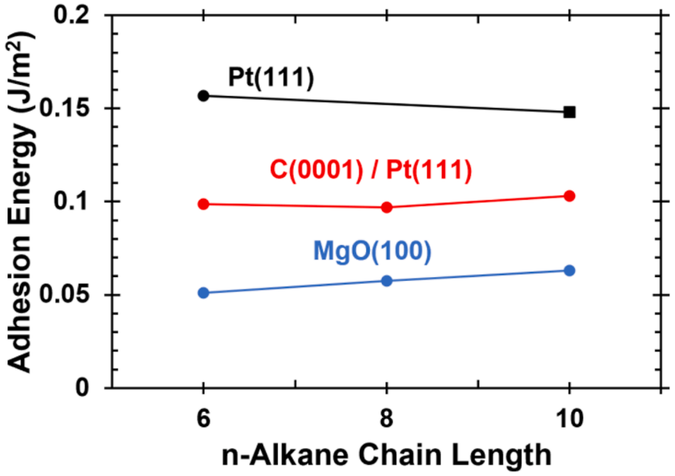

Fig. 2. Adhesion energies of liquid *n*-alkanes versus chain length on three surfaces, estimated from low-temperature measurements of heats of adsorption versus coverage using Eq. (1). Circular points were determined previously[12] using published TPD desorption energies,[13] while the square point is determined from the calorimetrically measured heat of adsorption of *n*-decane on Pt(111) using Eq. (1) in this work. The potential error bars on these points are estimated to be less than ~15% based on a complex set of contributions from both assumptions in Eq. (1) and experimental error bars in absolute heat and coverage calibration.

the same area per adsorbed alkane molecule.

This work now extends this understanding to systems that desorb irreversibly, by filling in the missing *n*-decane on Pt(111) adhesion energy. Not only do we see the expected independence of adhesion energy from chain length, but we also confirm that both TPD and experimental heats of adsorption can be used to determine the adhesion energies of bulk liquids on a surface. As there is no way to currently directly measure adhesion energies on clean metal surfaces, this indirect technique used in Fig. 2 has great value in providing new insights using previously measured heats of adsorption, or desorption energies, of solvent molecules at lower temperature.

5. Conclusions

The heat of adsorption of *n*-decane on Pt(111) versus coverage was measured using SCAC at 150 K. The initial heat of adsorption was nearly constant at 152 kJ/mol though 0.0455 ML (except for the first two points below 0.004 ML, which were ~17 kJ/mol higher and attributed to defect sites). The heat then dropped to ~90 kJ/mol by 0.091 ML, then decreased to the multilayer heat of adsorption of 84.9 kJ/mol above 0.27 ML. The integral heat of adsorption at the saturation coverage of the first layer of adsorbed *n*-decane (0.091 ML) was 132.7 kJ/mol. This was close to that predicted based on desorption energies measured by TPD for shorter *n*-alkane on Pt(111) (C_1-C_6), which were found by Tait et al.[13] to increase nearly proportional to carbon number.

Using the experimentally measured heat of adsorption versus coverage out to a bulk-like multilayer coverage, we estimate the adhesion energy of liquid *n*-decane on Pt(111) at room temperature to be 0.148 J/m^2. This is very close to the adhesion energy per unit area of *n*-hexane on Pt(111) estimated based on TPD desorption energies. Thus, linear alkanes have average heats of adsorption in close-packed islands on Pt(111) which approximately equal to a constant (13.3 kJ/mol per C atom) times their total number of CH_2 plus CH_3 groups, but because each CH_2 and CH_3 group occupies nearly the same area on the surface, the adhesion energy per unit area is also nearly a constant (0.15 J/m^2).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.susc.2022.122166.

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