An Experimental-Theoretical Study on Static Batch Sublimation with Laminar Flow and Constant Wall Temperature

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Abstract. *The major features of a static batch sublimation process over a hot plate with constant temperature were investigated in an experimental-theoretical study. An experimental apparatus with a real-time display was built to sublimate dry ice blocks of different sizes, in either circular or rectangular geometries. When temperature of the hotplate was changed from -30 to 200 °C, heat transfer coefficient "h_{sub}" decreased from 126 to 70 W/m²K, while thermal flux increased, linearly. Weight and area of the block had a positive/negative effects on heat transfer, respectively. In theoretical part, two "linear-gradient" and "cubic" models were developed by a combined mass-momentum-energy balance. The latter used Von Karman temperature profile, and in cases of circular and rectangular geometries could estimate "h_{sub}" with 17.8 and 13.5 % average error. Linear-gradient was analytic, with similar accuracy in the circular case. The developed model are especially useful for design of sublimation equipment in purification of the chemicals.*

Keywords: *Static sublimation, Heat transfer coefficient, Experimental and theoretical work, Analytical model, Laminar flow.*

1. Introduction. Sublimation is a phase-transitions process that occurs less commonly in the industry. It has certain applications in purification of valuable chemical (e.g. I_2) [1-3], gas feeding of the process lines [3], dye sublimation as a new printing method [4], heat pumps and refrigeration systems based on CO_2 [5-7], freeze drying [8], defrosting of the frozen surfaces [9], and recently as a new type of heat engine [10].

In industry, sublimation often occurs in one of the two following modes: 1-Batch (static) sublimation from a vessel containing the considered material, in which energy provided by an external heater is transferred to the solid substance via the conductive walls of the capsule [3, 11]. 2-Flow

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sublimation in which solid is sublimated by the heating effect of a flowing gas, via a coupled convection-mass transfer mechanism [5-7, 12-13]. A good example of the first case is found in uranium enrichment plants where the UF_6 capsules are heated in an oven to sublimate their content and condense it again for the sake of purification [3]. The second category, for instance occurs in a CO_2 refrigeration cycle that works under triple point of CO_2 (-56.6 ºC) [14].

In academy, work on sublimation process have been pursued on several research lines. The first line is molecular theory of sublimation that can be traced back to Maxwell [15]. Maxwell believed that a portion of the gas molecules that collide on a solid surface, is adsorbed and after a short period re-sublimated, while the rest part is elastically reflected. Smoluchowski tried to address detail of energy exchange between gas and solid molecules by defining coefficients of accommodation and slip [16-17]. Probably, Langmuir was the first scientist who focused on the sublimation process itself, and derived equations for its rate [18]. After Langmuir, and especially in the recent years, focus has shifted toward elucidation of the molecular theory of evaporation [19-23]. These new efforts are highly useful in developing molecular theory of sublimation, due to the close resemblance between evaporation and sublimation processes.

Another aspects of the sublimation that has been investigated thoroughly, is thermodynamic of the solid-gas equilibrium that has a long history, with numerous reports [24-27].

In comparison to the abovementioned research themes, heat transfer aspects of the sublimation process has been subject of fewer studies. Interestingly, most of the works in this area was related to the flow sublimation. Zhang and Yamaguchi [5], who were interested in developing low temperature CO_2-based refrigerators made major contribution in this field. They made a sophisticated experimental setup, and undertook both experimental and theoretical approaches [5-7]. Typical value of the heat transfer coefficient, reported by this group was around 300 W/m^2K.

Besides the aforementioned works, Sparrow and Spalding [28], Spalding and Christie [12], and some other authors [29-30] made notable contributions in mathematical modelling of the flow type sublimation. In their study, a channel whose insulated walls were covered with a sublimating material, was subjected to the hot stream of a flowing gas. The authors assumed a coupled mass transfer-heat transfer mechanism that can be regarded as generalization of the Graetz problems for pure heat transfer and pure mass transfer mechanisms.

In the field of batch (static) sublimation, an elegant contribution was made by Dayson who designed a new family of gas bearings based on sublimating CO_2 as the gas cushion [31]. The mathematical treatment of the subject by Dayson comprised several pivotal assumptions that were used in the current study. These include considering conduction through the gas layer as the main heat transfer mechanism, taking a laminar velocity distribution in the gap space, and eventually equating the weight of the shaft with the surface integral of the pressure. Unfortunately, Dayson's focus was on mathematical treatment of bearing design. As a result, no explicit formula was derived for the heat transfer coefficient (h_{sub}), nor was any experiment performed to support the discussions.

The Dayson's work can be principally regarded as revisiting of the theory of Leidenfrost effect, and its application in the sublimation process [32-35]. Leidenfrost effect occurs when a drop of liquid comes into contact with a very hot surface [32]. The initial vigorous boiling of liquid create a cushion of vapor, on which the droplet hovers the surface. Since this phenomenon is significant form both theoretical and practical standpoints, it has been very fully investigated by different researchers [32-35]. Correlations for heat transfer coefficient of the Leidenfrost boiling was suggested by several authors, among them, Berenson's equation (i.e. a modification of the Bromley's formula) is probably the most significant one [36].

The above examples show that in contrast to the flow type sublimation, lesser works have been done on batch or static sublimation. This is especially true about the heat transfer coefficient of the batch sublimation (h_{sub}), about which there is nearly no report in the open literature.

Batch (static) sublimation is not only important for its practical applications in design of industrial sublimating equipment, but also is interesting from a theoretical standpoint. When a dry ice block is heated on a hotplate, heat transfer causes sublimation of CO_2, and flow rate of the liberated gas that moves toward the perimeter of the block progressively increases. This makes the batch sublimation a variable-geometry, variable-flow hydrodynamic problem whose rigorous modelling by the Navier-Stokes equations is quite formidable, even in the laminar region. Higher heat flux makes the flow turbulent, and adds to the complexity, significantly.

The current study was aimed at undertaking an experimental-theoretical investigation on batch sublimation in laminar regime under the constant temperature boundary condition. In the experimental section, we studied sublimation of a block of dry ice (CO_2) on an adjustable temperature hotplate. Carbon dioxide was selected as a sublimating material because it is inexpensive, nontoxic, noncorrosive and readily available. Two different geometries of circular and rectangular were selected for the dry ice block, but most experiments were carried out on the former.

In the theoretical part, at first, by combining mass, momentum, and energy balances, an approximate analytical formula was derived for estimation of "h_{sub}". The temperature profile in this model was linear, and the model was similar to the Leidenfrost correlation, but with considerably higher accuracy. Then, other temperature and velocity profiles were checked, especially the one suggested by Von Karman in treating of the boundary layer theory [37]. This resulted in a model with slightly better accuracy.

2. Experimental

2.1. Apparatus. Figure (1) displays a schematic view of the constructed experimental apparatus. The dry ice block was put on a slab of copper with dimensions of 250 × 250 × 10 mm^3. To measure the momentary mass of the block, the copper plate was suspended on four strain gauge load cells with a combined weighing capacity of 10 kg and accuracy of ±1 g. The copper hotplate was heated from below by radiation from an adjustable electrical heater element, whose maximum power was 1.6 kW. The copper hotplate was precisely leveled, to prevent moving of the dry ice block during the sublimation. Temperatures of the upper and lower surfaces of the copper hotplate were measured by two thermocouples of type K that were connected to a real time data acquisition system. In addition, uniformity of the temperature of the hotplate, was checked by another thermocouple that was moved beneath the lower surface of the plate.

Since triple point of CO_2 occurs at 5.1 atm, all of the experiments were conducted at atmospheric pressure to warrant a pure sublimation process. During the sublimation, temporal variation of the mass of the dry ice block was recorded, and from which, the rate of sublimation was estimated.

Experiments in which temperature of the hotplate was less than or equal to 50 ^{o}C, were performed in a cold (refrigerated) room of -40 ^{o}C. The sizes of the room were 2 × 2.5 × 2.5 m^3, and it was primarily built for storing of the frozen foodstuff.

2.2. Experimental procedure. The experimental study was started first by calibration of the made apparatus to obtain a correlation between the power of the adjustable heater element, and the temperature of the copper hotplate. For this purpose, a dry ice block was put on the surface of the hotplate, and the hotplate was heated by energizing of the electrical heater. The power of the heater then was set on different values and time was given, to arrive at steady state temperatures. Such procedure was much time consuming, and in order to attain a specific equilibrium temperature, we had to repeat the experiments several times to get a reliable adjustment.

After obtaining the aforementioned power-temperature relation, the main experiments were started by the following procedure. Having switched on the electrical heater, a scarified dry ice block was put on the copper hotplate, and adequate time was given to attain the steady state temperature. At this point, the scarified dry ice block was quickly replaced by a new one, and monitoring of the mass of the block and time began.

In this work, two sets of the experiments were performed. One set with circular (cylindrical) dry ice block and another with the rectangular ones. Cutting of the block into the required shapes was performed by using hole saws attached to a drill in the case of cylindrical samples, and fine hand straight saw in the case of rectangular specimens. The cylindrical samples due to their symmetry, were better for modelling and comparison. Thus, most of the experiments were carried out in this geometry.

Three important parameters whose effects were investigated were: 1)- Temperature of the hotplate, 2)-Size (diameter) of the block, and 3)- Weight of the block.

Temperature of the hotplate was changed from -30 to 200 ^{o}C by adjusting both power of the electrical heater, and temperature of the surrounding environment. Indeed, for adjusting the hotplate temperature when it was in the range of -30 to 50 ^{o}C, experiments had to be undertaken in a cold (refrigerated) room of -40 ^{o}C.

Diameter of the worked circular sample was either 6 or 9 cm. Sizes of the rectangular samples were approximately 10 × 20 cm^2, but their weights were different. Two types of experiments were considered and run for the rectangular samples. In type (1), two larger sides (length) of the block were plugged by putting two fire clay bricks very close to the block to stop the gas flow, while in type (2) CO_2 generated by the sublimation, was allowed to escape from all sides of the rectangle (Figure S1 in supporting information).

In order to change parameter "weight" in the experiments. Test were run with one, two or more blocks that were superimposed on each other. Also, in some cases, a thick paper was put on a dry ice block to accommodate some metallic balancing weight. For the rectangular samples, also weight and temperature effects were investigated, but in the smaller ranges.

[Insert Figure 1 about here]

During the sublimation, radius (or length and width) of a dry ice block did not change noticeably, and only its height changed, and this change was proportional to the mass of the blocks (See explanation of S2 in supporting information file). As a result, density of the block remained constant, and no effect of crack, or expansion was observed in the experiments.

When the dry ice block started sublimation, a narrow gap was formed between the block and the hotplate that acted as a conduit for the sublimated gas. Measurement of the thickness of this gap (δ) was very formidable, because one had to avoid touching of the dry ice block, for not disturbing of its weight measurement by the load cells. Taking maximum possible care, and using a feeler gauge set, we could only obtain a coarse estimation of δ as $0.1 \leq \delta_{Aver.} \leq 0.5$ mm where subscript "Aver." should read average between all experiments.

3. Analytical Modelling.

3.1. General considerations. Based on the experimental observations, it was assumed that the dry ice block is suspended on a gas layer with thickness δ that is created by the sublimation process.

Our experimental observation revealed that in a typical sublimation test: (I) - Radius of the dry ice block (or width and length of rectangular samples) does not change, appreciably, and only its thickness varies with the time, (II) Convection heat transfer from sidewalls of the block is negligible, (III) - Sublimation surface density (g/cm^{2}s) is uniform in the bottom of the dry ice block, (IV) - Temperature of the hotplate surface beneath the CO_2 block is uniform, (V)- Radiation between the hotplate and the dry ice block can be ignored in comparison to the conductive heat transfer, and (VI) - Inside the dry ice block, temperature is uniform, and constant at -78 ^{o}C. This implies that the conduction heat transfer into the ice block is nearly zero.

Section S2 in supporting information provides justifications for the aforementioned propositions.

By following the method used for formulation of the Leidenfrost effect, and that of the Dayson [31,32], it was assumed that the principal heat transfer mechanism from the hot plate to the dry ice block is conduction through the CO_2 gas layer.

For derivation of an approximate analytical model, a two-stage procedure was undertaken. At the first stage, a viscous momentum balance was made, and at the second, energy balance was incorporated into the model.

Formulation of the model was based on a quasi-steady state assumption. However, the derived equations were then corrected by time-averaging over the period of the experiment. Reason for considering a quasi-steady state approximation was large difference between time scale of sublimation experiments that lasted about 1000 s, and time for gas to travel the radius of the CO_2 block which took only 0.03 s.

It is necessary to point out that the derivation was essentially concerned to the circular shape, where cylindrical symmetry simplified the mathematical treatment. In the case of rectangular geometry, an equivalent radius was defined, through which, the circular model was applied onto the experiments.

3.2. Mass and momentum balances. Figure (2) illustrates schematic view of a sublimating dry ice block on a hotplate under constant temperature boundary condition, along with the geometry of the gas conduit. Gas moves in the radial direction. The mass flow rate of the CO_2 gas, ($\dot{m}_{out}$) can be written as:

$$\dot{m}_{out} = \frac{\dot{Q}}{\Delta H_{sub}} \quad (1)$$

$$\dot{m}_{out} = 2\pi R\delta \rho_{gas} v_{out} \quad (2)$$

$$\sigma \pi R^2 = \dot{m}_{out} \quad (3)$$

where $\dot{Q}$ denotes rate of the heat transfer, ΔH_{sub} is latent heat of the sublimation, R shows radius of the dry ice block, σ is surface density (i.e. flux) of the sublimation, and eventually ρ_{gas} and v_{out} stand for density and velocity at exits of the gap, respectively.

[Insert Figure 2 about here]

Flow of CO_2 in the gas conduit was laminar. To justify this assumption, we calculated Reynolds number for this flow, for an average rate of the sublimation, and the maximum measured thickness of the gap space (i.e. $\delta \approx 0.5$ mm):

$$\left(\frac{\dot{m}}{\pi R^2}\right)_{Ave.} \approx 4.49\times10^{-3}\ kg/m^2 s \ \rightarrow\ v_{out,Ave.} = \frac{\dot{m}}{2\pi R\delta\rho_{gas}} \approx 0.95\ m/s$$

$$D_e = \frac{4A}{P} = \frac{4\times 2\pi R\delta}{2\times 2\pi R} \approx 2\delta \qquad (4)$$

$$\mathrm{Re}_{Ave.} \approx \frac{v_{out,Ave.} D_e \rho_{gas}}{\mu} = \frac{0.95\times100\times10^{-3}\times1.60}{14.84\times10^{-6}} = 102.4$$

where in definition of the hydraulic diameter (De), "A" and "P" denote passage area of the gas flow , and the corresponding wetted perimeter, respectively. Note that we used the maximum value of "δ" to make estimation of the Reynolds number more conservative.

For the laminar flow of the gas between two parallel planes with a progressively increasing flow rate, the momentum balance around the gas (Figure 2-b) can be written as:

$$-2\tau_w(2\pi r dr) - \frac{dP}{dr}\delta\,(2\pi r dr) = \rho_{gas}\frac{d}{dr}\left(\int_0^{\delta} 2\pi r v^2 dz\right)dr \qquad (5)$$

where τ_w denotes shear stress at surfaces of the CO_2 block and the hotplate.

The velocity profile in equation (5) is unknown, but by considering a laminar flow, and the fact that velocity vanishes at upper and lower surfaces, we arrive at:

$$v = Cz(\delta - z) \qquad (6)$$

where "C" only depends on "r", and shall be obtained from equation (3) in the following manner:

$$\rho_{gas} 2\pi r C \int_0^{\delta} z(\delta - z) dz = \sigma \pi r^2 \rightarrow C = \frac{3\sigma r}{\rho_{gas}\delta^3} \quad (7)$$

Shear stress at the upper and lower surfaces of the conduit can be calculated using the following formula [32]:

$$|\tau_w| = \mu \frac{\partial v}{\partial z}\bigg|_{z=0} = \mu \frac{\partial v}{\partial z}\bigg|_{z=\delta} = \mu C \delta \quad (8)$$

Combining equations (5), (6) and (8) will result in:

$$-2\mu C\delta - \delta \frac{dP}{dr} = \rho_{gas} \frac{1}{r}\frac{d}{dr}\left(rC^2 \int_0^{\delta} z^2 (\delta - z)^2 dz \right) = \rho_{gas} \frac{\delta^5}{30} \frac{1}{r} \frac{d(rC^2)}{dr} \quad (9)$$

By inserting “C” from equation (7), we arrive at:

$$-\frac{dP}{dr} = \frac{1}{\rho_{gas}\delta^2}\left(\frac{9\sigma^2}{10} + \frac{6\mu\sigma}{\delta} \right) r \quad (10)$$

Since at r=R, gauge pressure becomes zero (P=0); integration of equation (10) will result in:

$$P = \frac{1}{\rho_{gas}\delta^2}\left(\frac{9\sigma^2}{20} + \frac{3\mu\sigma}{\delta} \right)(R^2 - r^2) \quad (11)$$

In order to obtain “σ” and “v_{out}” from the above equation, it is necessary to equate surface integral of P to the weight of the dry ice block, and use the “$\sigma \pi R^2 = 2\pi R \delta \rho_{gas} v_{out}$”.

$$\int_0^R P\, 2\pi r\, dx = mg \rightarrow \frac{3\mu\sigma\pi R^4}{2\rho_{gas}\delta^3} + \frac{9\sigma^2 \pi R^4}{40\rho_{gas}\delta^2} = mg \rightarrow \frac{3}{20}\sigma^2 + \frac{\mu}{\delta}\sigma - \frac{2mg\rho_{gas}\delta^2}{3\pi R^4} = 0 \quad (12\text{-}1)$$

$$4\pi R^2 \rho_{gas} v_{out}^2 + \frac{3\mu\pi R^3}{\delta^2} v_{out} - mg = 0 \quad (12\text{-}2)$$

[Insert Figure 3 about here]

3.3. Energy balance.

The simplest temperature profile for the moving gas at any cross section of the conduit is the following linear function, which is obtained from the above-mentioned boundary conditions:

$$\left| \begin{array}{lll} z = 0 & T = T_W & (I) \\ z = \delta & T = T_S & (II) \end{array} \right. \Rightarrow T = T_W - \frac{\Delta T}{\delta} z \quad (13)$$

where T_W, and T_S stand for temperatures of the hotplate and dry ice surface, respectively, and $\Delta T = T_W - T_S$.

In writing of the energy balance across the gap space, it had better to use an averaged multiplication of temperature and velocity as the convective energy parameter:

$$\overline{VT} = \frac{\int_0^{\delta} V(z)T(z)dz}{\delta} \tag{14}$$

Using equations (6), and (13), the above integration yields:

$$\overline{VT} = C\delta^2\left(\frac{T_W + T_S}{12}\right) = \frac{3\sigma r}{\rho_{gas}\delta}\left(\frac{T_W + T_S}{12}\right) \tag{15}$$

For the cylindrical sublimation problem, the energy balance for an infinitesimal volume of gas in the "*r*" direction as shown in Figure (3) is written as:

$$\rho_{gas} C_P 2\pi r\delta\, \overline{VT}\Big|_{r+dr} - \rho_{gas} C_P 2\pi r\delta\, \overline{VT}\Big|_{r} - \sigma\, C_P 2\pi r T_s dr = -k\frac{\partial T}{\partial z}\Big|_{z=0} \times 2\pi r dr - \sigma \Delta H_{sub} 2\pi r dr$$

(16)

which after clearing gives:

$$\frac{1}{r}\,\frac{d(r\overline{VT})}{dr} = \frac{\sigma T_s}{\rho_{gas}\delta} - \frac{1}{\rho_{gas}\delta\, C_P}\left(k\frac{\partial T}{\partial z}\Big|_{z=0} + \sigma \Delta H_{sub}\right)$$

(17)

The above equation does not include dissipation of mechanical energy, as it is often negligible in comparison to the enthalpy terms.

The derivative in the left side of equation (17) can be calculated from equation (15) as:

$$\frac{1}{2}\,\frac{d(r\overline{VT})}{dr} = \frac{3\sigma r}{\rho_{gas}\delta} T_C \tag{18}$$

where T_C stands for $(\frac{T_W + T_S}{12})$.

The rate of heat transfer at the metal surface can be obtained from temperature profile:

$$-k\frac{\partial T}{\partial z}\Big|_{z=0} = k\left(\frac{\Delta T}{\delta}\right) \tag{19}$$

Combining equations (17) to (19) leads to

$$\sigma\left(6C_P T_C - C_P T_S + \Delta H_{sub}\right) = \sigma\zeta = \frac{k\Delta T}{\delta} \qquad (20)$$

where ζ stands for sum of the quantities within the left parenthesis.

Combining equations (12-1) and (20), we will get:

$$\delta = \left\{ \frac{3\pi R^4 \left(\frac{3}{20} (\frac{k\Delta T}{\zeta})^2 + \frac{\mu k\Delta T}{\zeta} \right)}{2mg\rho_{gas}} \right\}^{1/4} \qquad (21\text{-a})$$

$$\sigma = \frac{k\Delta T}{\zeta} \left\{ \frac{2mg\rho_{gas}}{3\pi R^4 \left(\frac{3}{20} (\frac{k\Delta T}{\zeta})^2 + \frac{\mu k\Delta T}{\zeta} \right)} \right\}^{1/4} \qquad (21\text{-b})$$

The sublimation heat transfer coefficient is directly related to σ through the following relation:

$$h_{sub} = \frac{-k \left.\frac{\partial T}{\partial z}\right|_{z=\delta}}{T_W - T_S} = \frac{\sigma\Delta H_{sub}}{T_W - T_S} = \frac{k\Delta H_{sub}}{\zeta} \left\{ \frac{2mg\rho_{gas}}{3\pi R^4 \left[\frac{3}{20} (\frac{k\Delta T}{\zeta})^2 + \frac{\mu k\Delta T}{\zeta} \right]} \right\}^{1/4}$$

$$\zeta = 6C_P T_C - C_P T_S + \Delta H_{sub}$$

(22)

3.6. Time-Averaging of heat transfer coefficient and gas properties. Nearly all involving parameters in equation (22), remain constant during the sublimation process, except the mass of the dry ice block. Thus, we can write:

$$h_{sub} = F\, m^{1/4} \qquad (23)$$

where "F" is a coefficient that comprises all physical parameters other than "m".

Using the standard method of the time-averaging, the mean value of the heat transfer coefficient $\overline{h_{sub,\mathrm{model}}}$ can be calculated as:

$$\overline{h_{sub,\mathrm{model}}} = \frac{\int_0^{t_{\exp}} h_{sub,\mathrm{model}}\, dt}{t_{\exp}} = \frac{\int_{m_{ini}}^{m_{fin}} F m^{1/4} \frac{dt}{dm} dm}{t_{\exp}} \approx \frac{4F(m_{fin}^{5/4} - m_{ini}^{5/4})}{5 t_{\exp}} \overline{(\frac{dt}{dm})}$$

(24)

where "m_{ini}" and "m_{fin}" denote initial and final masses of the dry ice block, and "t_{exp}" stands for period of the sublimation. Note that in derivation of equation (24), it was implicitly assumed that the temporal rate of sublimation is nearly constant, an assumption that is verified in Figure (4).

[Insert Figure 4 about here]

The "F" coefficient in equation (24) contains several terms corresponding to the CO_2 properties. Since temperature difference between the hotplate and dry ice block is substantial, and the moving gas layers in the laminar flow have different temperatures, appropriate types of averaging are required for truthful estimation of the gas properties in the gap. We made use the simplest type of the averaging as:

$$\langle Y \rangle = \frac{\int_{T_S}^{T_W} Y(T) dT}{T_W - T_S} \tag{25}$$

where "$\langle Y \rangle$" represents the average value of the parameter "Y". Note that in practice, T_W and T_S were constant throughout the sublimation area, and while T_S was constant at -78.5°C, the hotplate temperature T_W varied from an experiment to another.

For calculation of integral of equation (25), CO_2 properties were retrieved from References [39] and [40], and plotted against temperature. Then, the data were fitted with polynomial trend-lines and the trend-lines equations of high accuracy ($R^2 \approx 1$) were taken as Y (T). The following polynomial is an example of such fittings, and a complete list can be found in section S5 of supporting information file.

$$v_{CO2}(T) = 2.7166 \times 10^{-10} T^3 - 3.65791 \times 10^{-7} T^2 + 0.002040356\, T - 0.028138503 \qquad R^2 = 0.9999999$$

(26)

where T is temperature in Kelvin, and $v_{gas,CO2}$ is specific volume of the gas in one atmosphere. Using the above equation, average density of gas was found as:

$$\langle \rho \rangle = 0.87 \times \frac{\int_{T_S}^{T_W} \frac{1}{v_{CO2}(T)} dT}{T_W - T_S} \tag{27}$$

Coefficient of 0.87 in numerator of equation (27) shows pressure of the laboratory in atmosphere.

4. Results and Discussion.

4.1. Estimation of the experimental heat transfer coefficient. The experimental "h_{sub}" values were obtained from the sublimation rate by the following relation:

$$h_{sub,\exp} = \frac{\Delta H_{sub}\left(\frac{-dm}{dt}\right)}{A(T_W - T_S)} \qquad (28)$$

All quantities in equation (28), except "the rate of sublimation" were time-invariant; thus the averages value of experimental "h_{sub}" in Tables (1), and (2) was calculated by:

$$\overline{h_{sub,\exp}} = \frac{\int_0^{t_{\exp}} h_{sub,\exp} dt}{t_{\exp}} = \frac{\Delta H_{sub}\overline{\left(\frac{-dm}{dt}\right)}}{A(T_W - T_S)}$$

(29)

where t_{exp} is the period of the experiment; and the mean value of $\frac{-dm}{dt}$ was obtained from averaging of the tangential slope of the "t-m" curve, for each experiment (see Figure 4).

4.2. Behavior of the experimental data, and its comparison with the model. Tables (1) and (2) encompass the experimental conditions used for studying of the sublimation process in the case of circular block, along with the values of experimental and calculated heat transfer coefficients. As it is seen, data was categorized into two sets of "temperature effect" and "weight effect", each comprising of a few subsets.

For the aforementioned data that were gathered by repeating the experiments and averaging the data, an uncertainty analysis was undertaken and the results is recorded in Table S4 of the supporting information file. As it is seen, the maximum error is about $\pm$ **5**%.

Table S5 in supporting information file shows the experimental conditions used in sublimation of the rectangular ice block.

Figure (5-a) displays variations of the experimental "$\overline{h_{sub}}$" versus the hotplate temperature for two different diameters of the dry ice block. Evidently, heat transfer coefficient is larger for the smaller dry ice block, but in either size, it decreases with increasing of the temperature. Declining of the heat transfer coefficient with hotplate temperature is in agreement with the linear gradient model, because equation (22) contains temperature difference ($\Delta T = T_W - T_S$) terms in its denominator. Also, according to the aforementioned equation, $\overline{h_{sub}}$ is inversely proportional to the radius of the CO_2 block, and consequently tends to decrease with increasing of the diameter of the block.

Another effect, observed in Figure (5-a), is flattening of the T- $\overline{h_{sub}}$ curves at higher temperatures. This phenomenon will be addressed in the next paragraphs.

Besides examining of the behavior of " $\overline{h_{sub}}$ ", it is worthwhile to study the trend of variation of heat flux (i.e. $\frac{\overline{q}}{A} = \overline{h_{sub}}(T_W - T_S)$) as a function of the temperature. Figure (5-b) displays the T-q/A curves for different sizes of the dry ice block. The experimental heat fluxes present an obvious linear dependence on ΔT, and ascend with increasing of the hotplate temperature.

[Insert Figure 5 about here]

In order to elaborate the aforementioned effects in a more quantitative manner, the theoretical values of $\overline{h_{sub}}$ and $\frac{\overline{q}}{A}$ were plotted against the temperature by using equation (22) in Figure (6).

Likewise to the experimental data, the theoretical $\overline{h_{sub}}$ function descends with increasing of T_W, but it does not level off at higher temperatures (Figure 6-a). In attempt for understanding such discrepancy, we plotted the following "F_1" function, which is the main part of the $\overline{h_{sub}}$ formula (eq. 22), and it has an obvious dependence on temperature.

$$F_1 = \frac{k}{\xi}\left\{\frac{1}{\frac{3}{20}(\frac{k\Delta T}{\xi})^2 + \frac{\mu k \Delta T}{\xi}}\right\}^{1/4} \qquad (30)$$

[Insert Figure 6 about here]

The drawing of "F_1" in Figure (S6-a) of supporting information exhibits an asymptotic flattening behavior. However, the $\overline{h_{sub}}$ formula (i.e. equation 22), besides the F_1 term, contains the " $\rho_{gas}^{1/4}$ " factor. This latter term decreases with increasing of the temperature (Figure S6-b), and as a result, its multiplication with F_1 makes $\overline{h_{sub}}$ a monotonically descending function of the temperature.

The aforementioned explanations of the last paragraphs reveals that albeit the success of the linear gradient model in addressing the declining trend of $\overline{h_{sub}}$, it still needs more refinement to cover the leveling off behavior of the T- $\overline{h_{sub}}$ curve. Interestingly, the linear gradient model can very closely simulate the experimental trend of the elevation of heat flux versus temperature (Figure 6-b).

Figure (7-a) exhibit the experimental values of " $\overline{h_{sub}}$ " that were plotted against the mass of the dry ice blocks. The heat transfer coefficient increases with the mass of the block, and this effect is more appreciable for lower temperatures, and smaller sizes of the dry ice cylinder. According to equation (22), $\overline{h_{sub}}$ is proportional to the fourth root of the dry ice weight. Thus, in order to check ability of the model in simulation of the experiment, the experimental heat transfer coefficient was plotted against fourth root of mass in Figure (7-b). The good linearity of the depicted curves (i.e. $R^2 \approx 1$) demonstrates applicability of the developed model.

[Insert Figure 7 about here]

Here, it is worth mentioning that $\overline{h_{sub}}$ in equation (22) is not a function of $\frac{mg}{\pi R^2}$ or the weight-pressure of the dry ice block. Instead, it is correlated to $\frac{mg}{\pi R^4}$. This behavior is arisen from non-uniform distribution of the pressure beneath the dry ice block. Indeed, as the gas travels from center of the block to its circumference, its mass flow rate and thus its velocity increases, progressively. The driving force for accelerating of the gas is pressure difference between internal point "r" and peripheral point "R"; and variation of the velocity across the traveling domain "0-R" brings about a non- uniform pressure distribution, as a result.

Quantitative appraisal of the linear gradient model can be better performed by comparison of the calculated heat transfer coefficients with the corresponding experimental data in Table (1). For a more complete comparison, we included the theoretical " $\overline{h_{sub}}$ " values from the Leidenfrost model in the aforesaid table. The Leidenfrost model was essentially developed for boiling of small liquid droplets near a hot surface, and according to reference (32), can be expressed as:

$$h_{sub} = \frac{k\Delta H_{sub}}{\xi}\left\{\frac{2mg\rho_{gas}}{3\pi R^4\left(\frac{3}{20}(\frac{k\Delta T}{\xi})^2 + \frac{\mu k\Delta T}{\xi}\right)}\right\}^{1/4} \qquad (31)$$

[Insert Table (1) about here]

[Insert Table (2) about here]

Taking a quick glance at the results of Table (1), reveals the following significant points:

(I) In most of the cases, both linear gradient and Leidenfrost models overestimate the heat transfer coefficient, i.e.:

$$Often: \quad \overline{h_{sub,\text{model}}} > \overline{h_{sub,\exp}} \qquad (32)$$

Although mathematical interpretation of this inequality is formidable, it shows that if by considering any physical mechanism, rate of heat transfer reduces slightly, the accuracy of model increases appreciably.

(II) It seems that average errors of the model is greater for the case of smaller cylinder (i.e. 60 mm). Most likely, because in the larger size of the dry ice block, the velocity and temperature profiles are better established, and the experiments can follow the models more closely.

(III) Both models appeared less accurate in Table (2), or in other word in simulating of the weight effect. However, care must be taken in interpreting this result, because weight experiments were conducted at 100 and 200ºC where the relative error of the models is greater than the average.

Average relative absolute error (ARAE, %) in Tables (1) and (2) is defined as follows:

$$Average\ relative\ absolute\ \ error = \frac{\sum_{i=1}^{n} \left| \frac{\overline{h_{sub,\text{model}}} - \overline{h_{sub,\exp}}}{\overline{h_{sub,\exp}}} \right| \times 100}{n} \qquad (33)$$

where indices of “exp” and “model” indicate the experimental and calculated values of the heat transfer coefficients for every row of the Table (3), respectively, and “n” is the total number of the experiments. The average absolute errors of the linear gradient model for Tables (1) and (2) were 12.72 and 22.28 %, respectively. In the same circumstances, the Leidenfrost model brought about 16.70 and 31.10 % of ARAE. When the results of two tables were combined in Table (3), the linear gradient and Leidenfrost models resulted in 17.3 and 23.6 % errors, respectively. This demonstrates the slightly better performance of the linear gradient over the Leidenfrost model, even though, both seems fairly acceptable in the realm of the practical heat transfer engineering.

In the case of rectangular geometry, the equivalent radius “R_e” was defined as follows:

$$\pi R_e^2 = LW \qquad (34)$$

where L and W show length and width of the rectangles. Using the above definition, linear gradient and Leidenfrost models were applied on rectangular sublimation data, and the results were tabulated in Table S5 (in supporting information). As it seen, the results show the similar trends as the circular shape.

Regarding Table S5, some points are worthy of consideration: 1- Type (2) experiments in which gas allowed to move in all directions, shows slightly better heat transfer coefficient. 2- The effect of weight on the heat transfer coefficient was again more pronounced than that of the temperature. 3- Both linear gradient and Leidenfrost models presents higher error in the rectangular case, more likely because their formulation are not apt for asymmetric case of the rectangle. Interestingly, for this data, also linear gradient model brings about higher level of accuracy.

[Insert Table (3) about here]

4.2.1. Physical mechanism for declining of $\overline{h_{sub}}$ with temperature. Until here, the discussion was rather mathematical. Now, we try to obtain a mechanistic perception of the occurred phenomena. When temperature of the hotplate increases, the rate of sublimation increases. Rising $\frac{-dm}{dt}$, or in other word the output gas flow rate ($\dot{m}_{out}$) should either escalate v_{out} or δ. But, according to Bernoulli equation as the simplest momentum balance, "v_{out}" is essentially determined by the weight-pressure of the dry ice block, and since this weight is definite, v_{out} cannot increase limitlessly:

$$P = \frac{mg}{A} = \rho_{gas} \frac{v_{out}^2}{2} \qquad (35)$$

As a result, the only way to expel the produced gas by sublimation from the system, is to increase the gap thickness δ. Since the main heat transfer mechanism is conduction through the gap space, increasing "δ" significantly decreases the $\overline{h_{sub}}$ coefficient.

4.3. Improving the model. The simplest method to improve the accuracy of the developed model was to go to more complex temperature or velocity profiles. We chose to only focus on the temperature profile, because of a twofold reason: 1- Previous researcher who had worked out the similar phenomena, also used the parabolic velocity profile [31-32], 2- When, later, we employed other velocity profiles such as the following cubic formula, accuracy did not change, noticeably:

$$v = Bz(\delta^2 - z^2) \tag{36}$$

We exploited the following profile for the gas temperature, which was used by Von Karman in treatment of the boundary layer theory [37]:

$$T = az^3 + cz^2 + dz + e \tag{37}$$

Also by following the Von Karman method, boundary conditions were set as:

$$\left| \begin{array}{lll} z = 0 & T = T_W & (I) \\ z = 0 & \dfrac{\partial^2 T}{\partial z^2} = 0 & (II) \\ z = \delta & T = T_S & (III) \\ z = \delta & -k\dfrac{\partial T}{\partial z} = \sigma \Delta H_{sub} & (IV) \end{array} \right. \tag{38}$$

where condition (II) is arisen from vanishing both normal and parallel components of the gas velocity on the metal surface as described by Prantdl [37], and condition (IV) represents the uniform rate of sublimation across the dry ice surface.

Application of the boundary condition (38) in equation (17) resulted in:

$$T = \left(\frac{\Delta T}{2\delta^3} - \frac{\sigma \Delta H_{sub}}{2k\delta^2}\right)z^3 + \left(\frac{\sigma \Delta H_{sub}}{2k} - \frac{3\Delta T}{2\delta}\right)z + T_W \tag{39}$$

And equation (15) changed to:

$$\overline{VT} = C\delta^2\left(\frac{7T_W + 13T_S}{120} + \frac{\sigma \delta \Delta H_{sub}}{40k}\right) \tag{40}$$

$$-k\left.\frac{\partial T}{\partial z}\right|_{z=0} = -k\left(\frac{\sigma \Delta H_{sub}}{2k} - \frac{3\Delta T}{2\delta}\right) \tag{41}$$

Which eventually led to the following energy balance:

$$\frac{3C_P\Delta H_{sub}\delta}{20k}\sigma^2+\left(6C_PT_B+\frac{3}{2}\Delta H_{sub}-C_PT_S\right)\sigma-\frac{3k\Delta T}{2\delta}=0 \tag{42}$$

The above equation should be solved simultaneously with equation (12-1) to results in two unknown parameters δ and σ, and through the latter the h_{sub} according to the following relation:

$$h_{sub}=\frac{\sigma\Delta H_{sub}}{T_W-T_S} \tag{43}$$

The column entitled "$h_{sub,Cubic}$" in Tables (1) and (2), presents the results of calculation of heat transfer coefficient by the aforementioned model. As it is seen, the accuracy of the new model is better in Table (1), and worse in Table (2), than the "linear gradient" model. When two tables are combined, ARAE for the Cubic model became 17.83% that is slightly worse than the linear gradient one.

While the "linear gradient" closely rivals on the accuracy issue with the "Cubic" model in circular geometry, the latter presents an appreciably better performance in the case of rectangular shape (Table S7 in supporting information). Overall, the "Cubic" seems to be a more accurate model, in general.

The "linear gradient" has an evident advantage over the "Cubic" model that is its analytical form. In comparison, the Cubic model can only calculate the heat transfer coefficient, numerically.

5. Conclusion . The current study took a combined theoretical-experimental approach to elucidate major features of the batch (static) sublimation. The experimental results showed that heat flux and sublimation rate increase with increasing of the temperature of the hotplate, but heat transfer coefficient (h_{sub}) declines as temperature rises, and it levels off at higher temperatures. This behavior to some extent resembles the pool boiling beyond the DNB point, where the heat transfer coefficient declines due to the blanketing of the metal surface by the vapor [32, 36]. Indeed, the sublimation system, has a negative feedback with the rate of heating, which deters excessive rate of heat transfer and attaining very large value of Q and h_{sub}

$$\textit{Free motion of dry ice block in z direction}:\quad Q\uparrow\Rightarrow T_W\uparrow\Rightarrow\delta\uparrow\Rightarrow h_{sub}\downarrow \tag{44}$$

In the discussion section, mechanism of declining of "h_{sub}" with temperature was explained. However, another noteworthy point deserves elaboration in here. The dry ice block was free in our experiment to move in the vertical or "z" direction. Thus, when temperature increased, gas buildup caused lifting of the block and hence decreasing of the "h_{sub}". In cases where some lock or barrier inhibit vertical movement of the dry ice block, "δ" becomes constant, and very likely the heat transfer coefficient increases with the temperature. This condition may occur in a capsule of UF_6 that lies horizontally in the floor of an oven, and is heated.

$$\Pr ohibited\ motion\ of\ dry\ ice\ block\ in\ z\ direction: Q \uparrow \Rightarrow T_W \uparrow \Rightarrow \delta = cons\tan t \Rightarrow h_{sub} \uparrow \qquad (45)$$

Care should be taken in assimilating of the boiling and sublimation, as the two processes despite the abovementioned similarities have certain differences as follows. 1: Boiling is highly sensitive to the nature and roughness of the surface on which it takes place, while for sublimation, the type of surface seems to be immaterial. 2: Bubble formation as the principal step of the boiling is a stochastic process that proceed through nucleation-growth mechanism, and it occurs at several points, simultaneously; while sublimation take place uniformly, continuously and without any growth step on the surface.

Another interesting phenomenon, observed in this study is: The sublimation heat transfer coefficient "h_{sub}" hardly varies over the range of the examined parameters, so that the ratio between its maximum and minimums is 1.8.

A comparison of the results of the current study, with the data of Zhang et al. [6] reveals that "h_{sub}" in batch sublimation is about one-fourth (or less) than the similar value in the flow type sublimation (h_{sub} ≈310 W/m^{2}K). This is likely arisen from enhancement of the rate of sublimation, due to the mass transfer mechanism, in the flow type process.

Three other important finding of the present study were:

1- Heat flux almost linearly increases with increasing of the temperature in sublimation.
2- The weight effect is more influential in enhancing of heat transfer coefficient than temperature.
3- In the rectangular geometry, the similar behavior of "h_{sub}" was observed in regards to variations of the hotplate temperature, and the weight of the dry ice block. Models that were developed for the circular case, were also applicable for the rectangular block, but with lesser accuracy.

At the end, it is worthwhile to remind that still more refined hydrodynamic models are needed to bring about the required accuracy for the sublimation process. A plausible model of this kind shall define a new method for averaging of the viscosity and other physical quantities, to attribute a greater share to the colder regions of gas gap (i.e. near to the dry ice surface). The reason for this demand is: The gas layers that are in vicinity of the dry ice block, would become mixed with the just sublimated CO_2 gas, and their temperature approaches to the sublimation point.

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7. Data Availability. The data that supports the findings of this study are available within the article and its supplementary material.

8. Nomenclature

A	Surface area of the CO_2 block, m^2
b	Half-width of dry ice block, m
C	Coefficient of velocity profile in equation (6), $m^{-1}s^{-1}$
C_P	Heat capacity of CO_2 gas, J/kg K
D_e	Hydraulic diameter of gap space, m
g	Gravitational acceleration, m/s^2
h_{sub}	Heat transfer coefficient, Watt/m^2K
$h_{sub,exp}$	Experimental heat transfer coefficient, Watt/m^2K
$h_{sub,Model}$	Model value of heat transfer coefficient, Watt/m^2K
L	Length of the dry ice block, m
l	Half-length of dry ice block, m
M	Coefficient of equations (25) and (28), 1-dimensional, Watt/m^4
M'	Coefficient of equation (41), 2-dimensional, Watt/m^4
M''	Coefficient of equation (44), 2-dimensional, Watt/m^4
M'''	Coefficient of equation (45), 2-dimensional, Watt/m^4
m	Mass of dry ice block, kg
$\dot{m}$	Mass flow rate of gas at point x for 1-dimensional, kg/s
$\dot{m}'$	Mass flow rate of gas at point x for 2-dimensional, kg/s
$\dot{m}_{out}$	Rate of sublimation of entire block, kg/s
$\dot{m}^*$	Mass flow rate at one end of CO_2 block, 1-dimensional, kg/s
$\dot{m}^{*\prime}$	Mass flow rate at perimeter for 2-dimensional, kg/s,
N	Coefficient of equations (25) and (28), Watt /m
n	Number of experiments in a category, equation (61)
P	Gauge pressure, Pa
$\dot{Q}$	Total rate of heat transfer to the CO_2 block, Watt
S	Cross sectional area of the gap, m^2
SR	Sublimation rate, g/m^2s

T	Temperatures of gas at point z of gap space, K
T_S	Temperatures of the dry ice surface, K
T_W	Temperatures of the hotplate surface, K
t_{exp}	Period of the sublimation experiment, s
U	Coefficient of equations (25) and (28), 1-dimensional, Watt/m^9
U'	Coefficient of equation (41), 2-dimensional, Watt/m^9
U''	Coefficient of equation (44), 2-dimensional, Watt/m^9
U'''	Coefficient of equation (45), 2-dimensional, Watt/m^9
v	Velocity of gas, m/s
V	Velocity of gas, m/s
v_{CO2}	specific volume of CO_2 gas, m^3/kg
v_{out}	Exit velocity of gas at circumference of CO_2 block, m/s
w	Width of the dry ice block, m
x	Distance from centerline of the block in length direction, m
Y	Any physical property of CO_2 gas
<Y>	Average value of property Y with respect to temperature
y	Distance from centerline of block in width direction, m
z	Distance from hotplate surface, m

Greek Letters

α	Exponent of equation (64), dimensionless
β	Exponent of equation (47), dimensionless
γ	Exponent of equation (64), dimensionless
ΔH_{sub}	Heat of sublimation, J/kg
ΔT	Temperature difference between hotplate and dry ice, K
δ	Thickness of the gap space, m
ζ	Exponent of equation (64), dimensionless
η	Exponent of equation (64), dimensionless
κ	Exponent of equation (64), dimensionless
λ	Exponent of equation (64), dimensionless
μ	Viscosity of gas, Pa.s
ξ	Exponent of equation (64), dimensionless
ρ_{gas}	Density of gas, kg/m^3
σ	Surface density of the sublimation rate (kg/m^2s)
τ_w	Shear stress at surface of hotplate and the dry ice block, Pa

ψ	Exponent of equation (64), dimensionless
ω	Exponent of equation (64), dimensionless

Dimensionless groups

Pr	Prandtl number
Re	Reynolds number

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