



Preliminary investigation of the separation of a binary, two-phase hydrocarbon mixture using a commercially available vortex tube
by Neil Matthew Spracklen

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemical Engineering
Montana State University
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Abstract:

The vortex tube, a device which is customarily used to effect a temperature separation in an air stream, is being considered for use as a means to separate oxygen from air for potential use in the propulsion systems of hypersonic flight vehicles. For flight vehicle applications, it is desired that the device be capable of producing a liquid stream which is of at least 90 % oxygen purity, and which contains at least 50 % of the oxygen in the feed stream. Although the desired separation has not been achieved to date, the most recent studies have yielded 80-85 % enrichment with recovery in the 30-36 % range.

The fundamental objective of this preliminary investigation was to simulate the desired separation in a commercially available vortex tube, using a system of hydrocarbons as a surrogate for air. It was believed that by using the surrogate mixture in a bench-scale environment, we could come to a better understanding of vortex tube performance without the difficulties and expenses of operating in the cryogenic temperature regime that is required for air systems.

A bench-scale system was constructed, a mixture of 21 % cyclohexane and 79 % n-pentane was selected, and a parametric study was devised to evaluate the effect of feed conditions on vortex tube separation performance.

The desired separation was not achieved using the commercial vortex tube, a unit which was designed for air flow rates much higher than we were using in the bench-scale environment. Modifications were made to the equipment in an effort to compensate for the lower flow rates; however, these modifications did not improve attempts to achieve the desired separation.

All of the data seem to indicate that the best separations achieved in this study were only equivalent to one equilibrium flash stage. Many of the separations did not even match the equilibrium flash case, which suggests that the action of the commercial vortex tube used in this study produced mixing, entrainment, of other processes which serve to reduce the separation when compared to that of an equilibrium flash.

PRELIMINARY INVESTIGATION OF THE SEPARATION OF A BINARY,
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This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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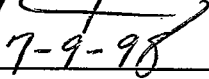
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ABSTRACT

The vortex tube, a device which is customarily used to effect a temperature separation in an air stream, is being considered for use as a means to separate oxygen from air for potential use in the propulsion systems of hypersonic flight vehicles. For flight vehicle applications, it is desired that the device be capable of producing a liquid stream which is of at least 90 % oxygen purity, and which contains at least 50 % of the oxygen in the feed stream. Although the desired separation has not been achieved to date, the most recent studies have yielded 80-85 % enrichment with recovery in the 30-36 % range.

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CHAPTER 1

INTRODUCTION

Project Framework

Since the early 1960's, there has been considerable interest in developing a system for hypersonic flight vehicles which could eliminate the necessity of carrying large quantities liquid oxygen from takeoff. One such concept employs utilization of an air collection and enrichment system, whereby a two-stage air-breathing vehicle collects air while traveling to orbit, eliminates most of the nitrogen component of the collected air, and selectively stores liquid enriched air which is about 90% oxygen. The ability to acquire en-route the necessary amount of liquid enriched oxygen to be used in the second stage of propulsion (rocket mode) beyond the air-breathing envelope can cut the takeoff weight in half, allowing the vehicle to carry a much greater payload (Maurice 316).

Project Overview

Efforts are currently being made to develop a system in a space vehicle which is capable of separating and storing a liquid oxygen enriched product stream from air collected en-route to orbit. Our specific research interests focus on the actual process which will effect the separation.

Phase One of this research project, completed in 1997, included an evaluation of various air separation technologies for use as air collection and enrichment systems (hereafter, ACES) on board space vehicles which utilize air-breathing rocket boosters for propulsion during the first stage of flight. Many air separation technologies were considered, but it was concluded that most conventional air separation technologies would be prohibitively large in volume and weight to be efficiently used in hypersonic flight (Binau 141-42). However, the Phase One study suggested that a system which employs the principle of combined differential condensation and differential vaporization has the potential to carry out the separation.

A device which may employ the combined processes of differential condensation and differential vaporization is the vortex tube, in that the overall process occurring in the vortex tube involves partial condensation and re-vaporization of a partially condensed feed. Although this device shows promise with respect to equipment weight and volume considerations, experiments to date have not achieved the desired separation of 90 % oxygen enrichment and 50 % oxygen recovery. The most recent tests by Balepin yielded 80-85 % oxygen enrichment with recovery in the 30-36 % range (Air Collection Systems 417).

Based on Balepin's findings, Phase Two of this project, currently under way, is an attempt to discover whether the desired separation is attainable using a vortex tube.

Project Goals

The fundamental objective of our portion of Phase Two was to model the separation of air in a vortex tube, with the aim of achieving a minimum separation of 90% O₂ purity and 50% O₂ recovery. Subsequent chapters of this paper address the three goals outlined to meet this objective:

1. Carry out an experiment on a commercially available vortex tube using a surrogate feed mixture of hydrocarbons which would complement the cryogenic investigations using air being carried out by MSE Technology Applications, Inc. of Butte, MT.
2. Through the use of a surrogate hydrocarbon system operating at reasonable temperatures, come to a better understanding of the apparatus and its potential for separation without the expense of the equipment and control difficulties associated with a system operated at cryogenic conditions.
3. Prepare a report on the findings of our investigation.

CHAPTER 2

BACKGROUND

Literature Review

Context of the Project

A widely agreed upon principle in the literature is that for a viable launch vehicle program to exist, it is an absolute economic necessity to be able to produce a vehicle which allows for its major components and systems to be recovered and reused on a continuous basis. Other economic factors which tend to affect the continued development of launch vehicles include a further reduction in overall vehicle cost and an increased payload capacity (Czysz and Murthy 582).

By and large, it is the propellant (oxidizer and fuel) and the propulsion system (rocket, airbreathing engine, or some combination of the two) which not only dictate the complexity of the control systems, the velocity limits and range of the vehicle, and the demands on materials and structures, but also are responsible for a significant and sometimes prohibitive portion of the total initial mass of the vehicle (Balepin et al. 1). Further, the selection of propulsion system determines whether the vehicle is capable of horizontal take-off and horizontal landing, which influences recoverability, flexibility, and facility costs.

In light of the influence the propellant and propulsion system has on the economics and the performance of a hypersonic space vehicle and its associated flight program, it can be proposed that successful and sustainable hypersonic vehicle design will depend on development and advances in propulsion-propellant technology (Czysz and Murthy 582). Thus, one of the great challenges facing developers lies within the task of creating a system which is optimally suited to meet air collection and separation requirements and, at the same time, minimizes equipment volume and mass.

Prior Studies of Air Separation Apparatus

Proposals and studies of potential ACES equipment with bias towards use on a hypersonic flight vehicle have been investigated for nearly four decades. While it is not within the scope of this paper to thoroughly account for all of the previous conclusions and selections made, it is perhaps useful to briefly review some of the more conventional separation technologies which have been considered, along with the conclusions made regarding their prospective use in space flight. Also, an abbreviated account of the conclusions drawn from Phase One of the current project will be presented.

Between 1959 and 1967, considerable effort was made to evaluate potential ACES concepts, largely through United States Air Force funding. In the first few years of this period, many conventional land-based air separation technologies were considered, such as co-current spray contactors, nitrogen freeze-out, centrifugation, molecular sieves, membranes, fractional distillation, vortex tubes, and chemical reaction (Maurice, Leingang, and Carreiro 318). The systems were evaluated with a bias toward their

respective potential of meeting a requirement that the involved equipment mass be very small when compared to the mass of the liquid enriched air (LEA) separated and stored during the collection phase. The conclusion was made in 1961 that the processes of fractional distillation and chemical reaction were the best candidates for meeting this requirement.

The chemical reaction concept considered utilizes the peroxide reaction. Barium oxide or cobalt oxide is used to extract oxygen from air at high temperature and pressure, forming a peroxide. The mixture is then subjected to a lower pressure which induces the peroxide to release oxygen and revert to the metal oxide. On the other hand, the fractional distillation concept employs a vapor phase being bubbled through successive stages of counter-flowing liquid, promoting oxygen enrichment of the liquid and nitrogen enrichment of the vapor. While the chemical reaction air separation concept seemed to be attractive on the basis of equipment weight and product recovery capabilities, it was eventually abandoned due to the mechanical complexities associated with the extremely high temperatures created by the highly exothermic oxygen extraction reaction. Consequently, a majority of the ACES research from that point on has focused on the development of various schemes of fractional distillation.

Cryogenic fractional distillation as a process to separate oxygen from air has been utilized in land-based systems for many years. While a much more detailed description of the process can be found in the literature (McCabe and Smith; Perry; Leingang, Maurice, and Carreiro 344-54), the general framework for fractional distillation for use in ACES involves the utilization of two distillation columns, one operated at high pressure

(near five atmospheres) and one operated at low pressure (atmospheric pressure). The feed mixture is compressed and fed into the bottom column (the high pressure column). The vapor stream from the top of this high pressure column is enriched in nitrogen, and is condensed by exchanging heat with the oxygen enriched liquid stream from the low pressure column in the reboiler-condenser, which is located between the two columns. This exchange further enriches the liquid stream from the low pressure column in oxygen, and as a result, product of high purity liquid oxygen is recovered. The vapor stream from the high pressure column is then returned as feed to both the high and the low pressure columns. High purity nitrogen vapor is recovered from the top of the low pressure column. Hydrogen is used to provide refrigeration in the reflux condenser for the low pressure column.

Although the weight and volume of the land-based system was proven to be prohibitively large for use in hypersonic flight vehicles, investigations in the 1960's revealed modifications to the land-based system which had a significant impact on the weight and volume (Leingang, Maurice, and Carreiro 344-354). The flat distillation trays in the land based unit were replaced with rotary high-g separator cylinders which rotate around their central axis (Nau & Campbell; Bonnet). The centrifugal force created by the rotating cylinder-trays induces the liquid in the system to travel in a crossflow pattern from tray to tray towards the outside of the column. Cylinder rotation also promotes higher vapor velocities, which allows for tighter tray spacing, a significant reduction in equipment size, and operation on a flight vehicle which experiences variable gravitational forces.

As a result of the modifications, both the weight and the volume of the columns in the unit were reduced enough to warrant the construction and testing of a full-scale system for use in hypersonic flight. The tests were quite successful in terms of product purity and recovery, and further, the project was validated in terms of meeting hypersonic flight vehicle weight and volume requirements (Leingang, Maurice, and Carreiro 350). Despite the successful test project, there is nothing available in the literature to suggest the rotary high-g fractional distillation concept has been further explored for use in hypersonic flight applications.

Phase One Investigation

Mentioned in the introduction, Phase One of the current research project was completed in 1997. The goal of the Phase One study was to screen available land-based air separation technologies for potential use in hypersonic flight vehicles. Technologies considered during this study included the following: silver, mixed conducting, polymeric, and facilitated transport membranes; reversible chemical reactions with molten salts, solid metallic oxides, and transition metal complexes; solid state adsorption; vapor liquid equilibrium; and magnetic fields.

One fundamental critical criterion used for screening was that any potential technology had to meet weight and volume requirements. Specifically, the mass of the system divided by the inlet air mass flowrate could not exceed 10 lbs/(lbs/s), and the volume of the system divided by the inlet air mass flowrate could not exceed 0.5 ft³/(lbs/s). With a given feed rate of 100 pounds per second to the separation system, this

meant the system had to weigh less than 1000 pounds, and occupy a space less than 50 cubic feet.

In his 1997 report on the Phase One study, Binau concluded that most of the traditional land-based air separations considered would exceed the weight and volume limits. However, it was suggested that certain aspects of vapor-liquid equilibrium technologies seemed to hold the most promise in terms of development of an air separation system which would meet the requirements for this application (Binau 141-42).

Rotary distillation, as described in the previous section, was found to be a vapor-liquid equilibrium system with possibilities. Considering that the original test system was constructed of aluminum and stainless steel (Leingang, Maurice, and Carreiro 346), Binau suggested that by utilizing lower density materials, such as carbon-fiber composites, a significant reduction in weight could be realized (114-15). Thus, the rotary distillation design might deserve further consideration.

Other vapor-liquid equilibrium technologies which Binau felt deserved further consideration included those processes which operate under the premises of differential vaporization and differential condensation (*vide infra*). One system which is thought to operate as a differential vapor-liquid equilibrium separation process is the vortex tube (Binau 140).

Differential Condensation and Differential Vaporization

Like other vapor-liquid equilibrium processes, differential separations exploit the compositional difference between the vapor and liquid phases that a non-azeotropic, multicomponent mixture will possess when it exists in the two-phase regime.

In a differential separation process, small amounts of a multicomponent feed stream are removed and segregated in a fashion which prohibits mixing or mass transfer between the two resulting (product) streams. The two general categories of differential separation processes are differential condensation and differential vaporization.

In differential condensation, the stream consisting of the removed product, or liquid tails, is depleted in the more volatile component. What is left of the original feed stream, or vapor heads, becomes increasingly enriched in the volatile component as it proceeds through the condenser. Differential vaporization operates similarly, except that the vapor heads stream being removed from the feed is enriched in the more volatile component, while the residual liquid tails becomes increasingly depleted in this component.

Viewed another way, differential condensation allows for some enrichment and high recovery of the more volatile component in the vapor heads, while differential vaporization allows for high enrichment of this same component, but at a lower recovery. Thus, the individual differential processes demand a tradeoff between enrichment and recovery.

McCandless, in his investigations of separating oxygen from air, has shown that a combination of the two processes allows for high recovery coupled with very high

degrees of enrichment (Air Enrichment I 20-22). In these investigations, for moderate relative volatilities, both enrichment and recovery were greater than 95 %. Figure 1 shows a simplified schematic of a recycle process which involves differential condensation followed by differential vaporization of the liquid tails stream.

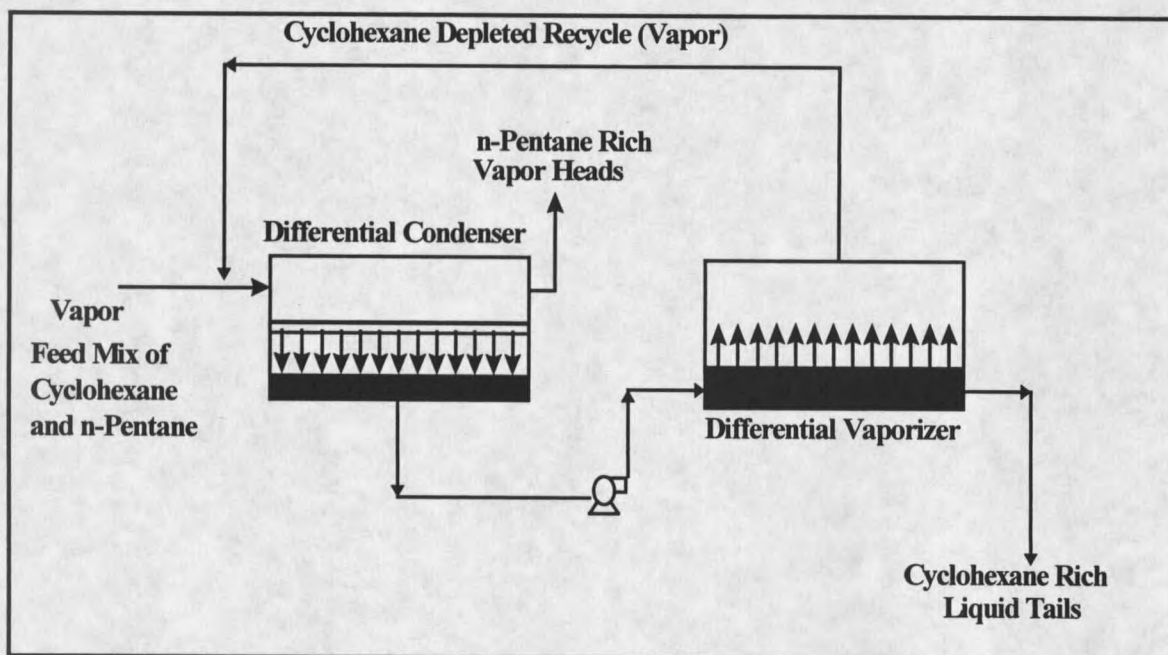


Figure 1. A combined differential separation process with recycle

Furthermore, when considering the separation of oxygen from air, both Binau (120-23) and McCandless (25-30) have suggested that, when compared to either ordinary distillation or flash condensation followed by flash vaporization, the combined process of differential condensation followed by differential vaporization with recycle is theoretically more efficient in terms of stages required for separation. Consequently a more compact system can be developed.

The exhaustive literature search in Phase One of the current study resulted in the conclusion that the overall process of differential condensation followed by differential vaporization is favored over any other process to meet the goals of the project for separating oxygen from air (Binau 131-32). However, while differential separation processes are attractive from a theoretical standpoint, development of hardware to carry out a combined differential separation process would be very difficult, due to the requirement that the discrete amounts of product removed from the feed stream must not be allowed to accumulate prior to removal, and sections of the feed stream, which are at different stages of composition, must not be allowed to mix or escape with the removed product stream (Binau 123-24).

As will be discussed in the next section, it is believed that the mechanism for separation in a device known as the vortex tube is a combination of processes which approach that of differential condensation coupled with differential vaporization.

The Vortex Tube

History. First conceived by Georges Ranque in the early 1930's, the counter-flow vortex tube is a mechanically simple device which separates a high pressure gas stream (typically air) into two product streams, one hotter and one cooler than the feed stream temperature (Ranque 1-6). The simplicity of the device lies in the fact that no mechanically moving parts are required to effect the stream separation. It is reported vortex tubes can produce streams with temperatures ranging from -40°C to 121°C ,

depending on the fraction of air released from the "hot" end, and subject to energy balances (ITW Vortec® Corp).

While the concept of replacing conventional refrigeration or heating systems with such a simple and compact device may appear attractive initially, it is well known that the vortex tube operations are thermodynamically inefficient (Bruno 988). A ratio of cooling achieved to compression energy required reveals the efficiency of this device to typically range from 10 to 15 % for air systems (Hadjik et al. 77). As a result, applications for the vortex tube have mainly been limited to those circumstances where small size, light weight, or intensely focused spot heating or cooling is required (Young and McCutcheon 522).

Operation and Theories. A cross-sectional schematic of a typical vortex tube configuration is given in Figure 2.

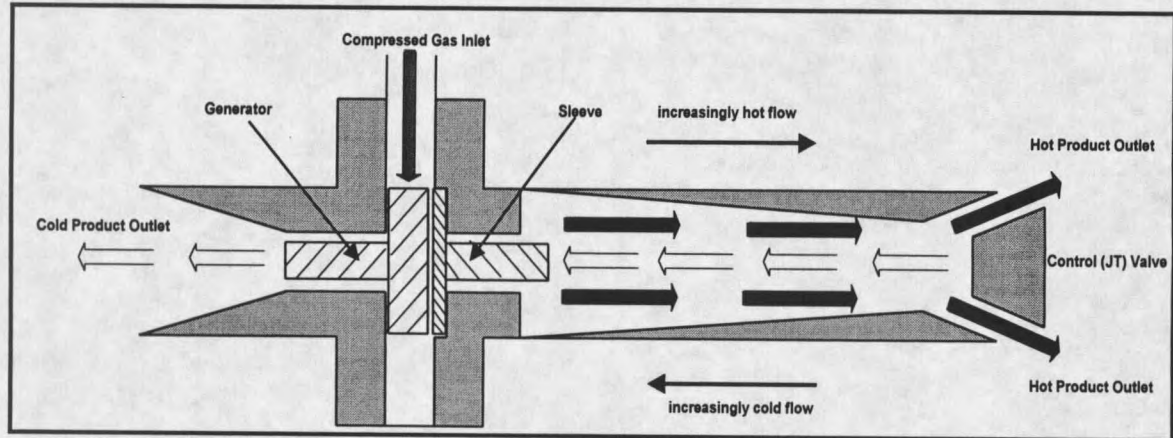


Figure 2. Schematic diagram of a typical vortex tube.

In the customary usage of the vortex tube, a source of compressed air is directed to the inlet nozzle, and passed through a vortex generator and sleeve. Once through the generator and sleeve, the gas is fed tangentially into the vortex tube, which is maintained at a low (near atmospheric) pressure. Introduction to the low pressure causes the gas to expand, and consequently, speed up to near sonic velocity (Bruno 987). As a result of the tangential introduction, a vortex is created, and as Craze notes, "the vortex thus formed creates an intense centrifugal field within which gas dynamic transport processes and to a lesser extent Joule-Thompson (JT) cooling establish temperature, pressure, and compositional gradients in the tube both axially and radially" (Craze, lines 24-28). A control valve at one end of the tube allows some of the gas to leave as hot product, while

