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Existing Biorefinery Operations That Benefit From Fractal-Based Process Intensification

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Abstract

Ion exchange, adsorption, and chromatography are examples of separation processes frequently used in today's biorefineries. The particular tasks for which these technologies can be successfully applied are highly influenced by capital cost and efficiency. There exists a potential for significantly increasing the efficiency of these processes whereas simultaneously decreasing their size and capital cost. This potential for process intensification can be realized with the use of engineered fractal equipment. The cost savings potential and the possibilities for broadening the use of fractal-based separation technologies in future biorefinery concepts is illustrated by examples of full-scale implementation in the sugar and sweetener industries.

Index Entries: Biorefinery; fractal; liquid separations; chromatography; ion exchange.

Introduction

In the past, agriculture was viewed as an industry providing single commodities such as grain, sugar, seed, and so on. For the past several decades agriculture has been slowly transforming into an industry processing agricultural feedstock into a wide range of value-added fuels and chemicals. An analogy can be made with petrochemical refining, in which oil is converted into various chemical products. Conventional corn processing can be used as an example of a biorefinery producing ethanol as a fuel, dextrose as base chemical, fructose syrup as a food sweetener, oils, vitamins, and complex animal feed. Global market conditions and competitive pressure are stimulating diversification in other agriculture-based industries, such as sugar, forestry, pulp, and paper. Additionally, a new industry is evolving, in which agricultural waste products, such as straw or other sources of inexpensive biomass are being used as raw materials for obtaining fuels and chemicals. The multicomponent nature of agricultural feedstock and the variability of its composition related to harvest and storage conditions make the task of creating a universal processing technology quite challenging. However, many lessons can be learned from

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operation of existing biorefineries. Because certain similarities exist between plant-derived feedstocks, analysis of unit operations involved in current processing technologies provides a good start for evaluating process and equipment design for the emerging bioindustry.

Innovative concepts, such as process intensification, will favorably impact the economics of future biorefining. The term "process intensification" was first introduced by ICI (UK) in the late 1970s. Creation of new types of equipment with much higher efficiencies and smaller footprint compared with conventional designs is the main focus of process intensification efforts. Resulting reduction of capital and operating cost allows reconsidering use of technologies currently deemed marginally feasible for certain applications. Henderson *(1)* indicates, however, that the advantages extend beyond capital cost issues and additionally provide improved process performance, lower safety risks, and improved manufacturing capabilities.

Considering the complex nature of plant-derived materials, it is likely that affinity-based separation technologies capable of isolating and purifying components with similar physico-chemical characteristics will be used extensively in the future biorefineries. With the emphasis on the fact that separation processes account for 40–70% of both capital and operating costs in the existing industry Humphrey *(2)* introduced a term "maturity" of a separation technology to measure the relative levels of fundamental knowledge and practical applications. The maturity values for affinity-based technologies, such as ion exchange, adsorption, and industrial chromatography range were estimated at 20–30%, whereas for more conventional crystallization and distillation processes the values exceed 65%. Therefore, there is a definite need for technology development efforts on all levels starting with a better understanding of fundamentals and optimization of materials, equipment, and modes of operation. The purpose of this article is to illustrate how the concept of engineered fractals as used in today's biorefineries can benefit the emerging renewable feedstock processing industry.

The Importance of Liquid Separation Technologies

A generalized sequence of unit operations common to existing biorefineries is shown in Fig. 1. Although the flow diagram does not include multiple recycle streams and handling of waste streams, it clearly demonstrates the importance of separation technologies (especially liquid separations). The purpose of the following discussion is to illustrate the similarities between two existing biorefinery operations—corn wet milling and sugar processing.

Technologies recovering valuable components from an agricultural feedstock always include a step for cleaning and preparation, in which a large uniform surface area is created to facilitate subsequent chemical/biochemical reactions or mass transfer. Grinding corn or sugar cane stalks and sugar beet slicing are examples of such preparation steps. The prepared biomass is then subjected to initial fractionation or extraction of major

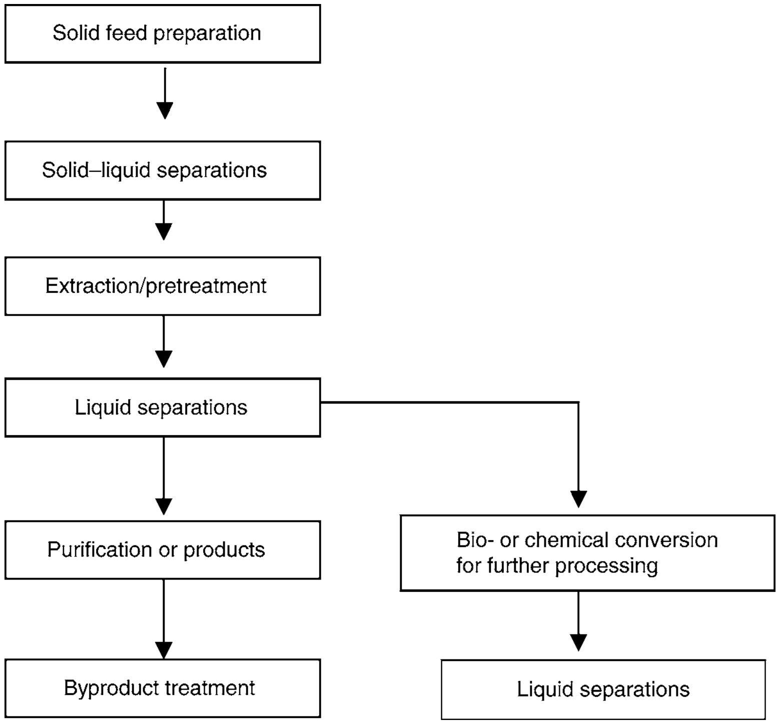

Fig. 1. A generalized sequence of unit operations in a biorefinery.

components, such as starch/gluten separation in the corn industry or a diffusion step in the cane or beet industry. Biochemical conversion into a new product in the corn wet milling process or chemical purification of sugars in beet processing are typically followed by ion exchange processes such as demineralization or softening, respectively. Examples of liquid separation technologies (adsorption, large-scale chromatography, and ion exchange) for downstream isolation and purification of products include enrichment of fructose syrup and polishing/decolorization of high-fructose corn syrup (HFCS) in the corn wet milling process and recovery of sugar from molasses and decolorization in cane refineries. These examples demonstrate the dominating role of liquid separation technologies in existing biorefineries. Out of 15 steps of HFCS production in a wet milling process five steps utilize ion exchange resin technologies for adsorption, fractionation, and purification *(3)*. This is not surprising, considering that the described methods are very efficient for separating sugars from organic acids, salts, and other impurities. Even separation of various sugars with similar properties is relatively easy to accomplish. Separation of glucose and fructose using simulated bed chromatography is a good example of a commercially accepted process that achieves high purities and recoveries of both products *(4)*.

Future biorefinery applications will take advantage of the technical solutions already developed in these example industries. It is anticipated,

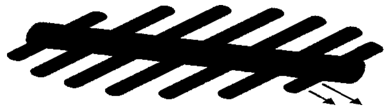

Fig. 2. An example of a conventional distributor.

however, that additional developmental efforts will be needed, including in the area of process intensification, to improve feasibility. An example of such process intensification in the area of fluid distribution in the industrial equipment is considered later.

Distributors Based on Fractal Geometry

Most common distributors used in chromatographic, softener, decolorization applications are quite simple in design. The major drawback of common types of distributors roots in the lack of symmetry, which makes a scale-up task very complicated. An example of a conventional lateral pipe distributor is shown in Fig. 2. Typically the centrally located inlet is connected to a series of interconnected pipes with outlet holes. The residence time from the inlet to each hole varies depending on the distance from the inlet. For the same reason the pressure drop is different for each path from an inlet pipe to an outlet hole. Because the concentration of dissolved components often changes continuously, different residence time leads to spreading or "smearing" of the concentration front and therefore to loss of separation efficiency.

Conventional practice of distributor design based on high-pressure drop or variable outlet hole size leads to another set of problems. Among them is dependence of distribution quality on the feed rate. This problem is sometimes addressed as a turndown ratio. The ability of a distributor to convert fluid from a feed pipe into a uniform two-dimensional surface inside the column is extremely important for many industrial applications. For example in simulated moving bed (SMB) chromatography recirculation flow varies significantly during an operation cycle. Obviously, variable distribution quality in each step results in deviation from plug flow conditions and, hence, reduction of overall process efficiency.

A new generation of fluid distributors based on fractal geometry has been described extensively in the recent literature *(5,6)*. Fractals can be defined in a number of ways. The following simple qualitative definition can be used to easily recognize such structures—"Fractals are self-similar objects whose pieces are smaller duplications of the whole object." Fractals can be "geometric" such that each iteration results in smaller and exact reproduction of the geometry (e.g., shapes in Fig. 3). In a separate category, "statistical" fractals are only self-similar in a statistical sense. An example is a tree in Fig. 3 (branches similar to the trunk, twigs similar to branches, and so on). The idea of application of engineered fractal for process applications has resulted in a number of patents on fluid distributors and

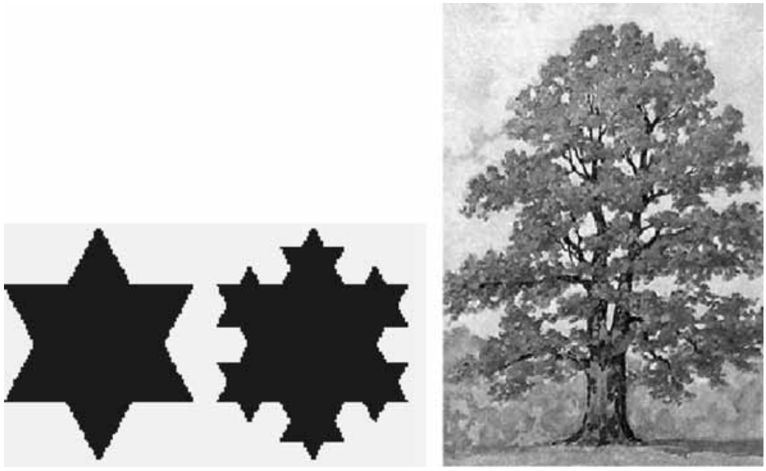

Fig. 3. Examples of geometrical (shapes) and statistical (tree) fractals.

engineered fractal cascades *(7,8)*. Fractal distributors originally installed in large-scale chromatographic columns (up to 7 m in diameter), have been modified for ion-exchange and decolorization applications, air distribution in conditioning silos, gas–liquid systems, and so on. A sample illustration of a fluid distributor for industrial chromatographic column up to 7 m in diameter is shown in Fig. 4. Fluid enters the predistributor designed to provide hydraulically equivalent flow to the fractal plates that are located on the top or bottom of the vessel. The final outlets provide *I* nearly ideal distribution quality. Each final outlet point is geometrically and hydraulically equivalent to the others, ensuring very uniform fluid distribution across column cross-section. Seemingly complicated, the distributors are rather easy to manufacture and install. The original distributors have been in industrial operation for more than 9 yr. Their cost compares favorably with conventional designs.

Process Intensification Using Fractal Technology

Related to fluid scaling and distribution methods evolved in nature (such as the human circulation system), engineered fractals exhibit unique features beneficial to the efficient operation of process equipment. They are especially efficient in the technologies that are sensitive to maintaining the narrow concentration front such as adsorption, ion exchange, distillation, sparging, mixing, and so on *(9)*. Engineered fractals were first applied in the industrial SMB chromatographic columns in 1992. Most important features of fractal devices are discussed later.

1. Fractals provide uniform fluid distribution to a surface or within a volume. This improves process efficiency in reaction vessels or mass transfer devices. As one example, fractals provide plug flow in

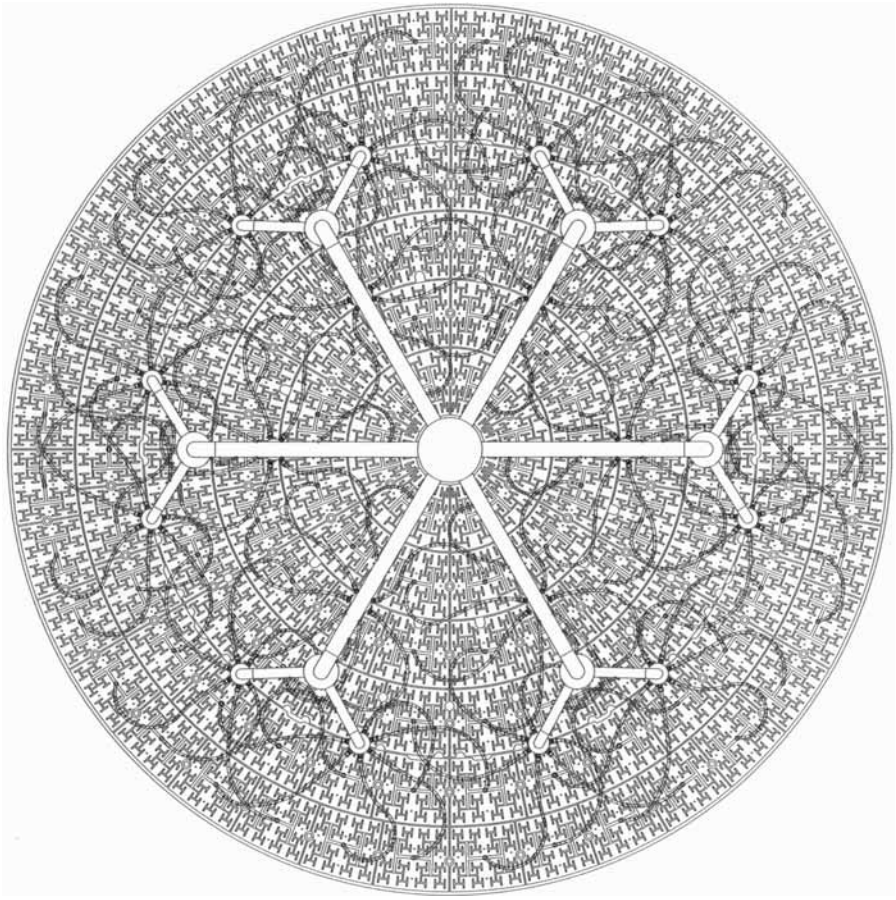

Fig. 4. Fractal distributor layout.

large-scale ion exchange or chromatographic columns. This results in significant savings in both capital and operating costs.

2. Fractals can be reliably scaled from small pilot equipment to large industrial equipment. The key to reliable scale-up is providing the same density and size of fluid inlets/outlets in both the pilot and subsequent industrial equipment. Thus, the hydrodynamic conditions for the fractals can be exactly reproduced. Construction of larger equipment is accomplished by adding larger fractal channeling to the initial pilot scale layout. Such scaling procedures have been validated for commercialization of large-scale chromatographic processes with columns up to 7 m in diameter. In these cases, the original pilot testing was performed using 3-in. diameter columns.
3. Fractals exhibit a very low sensitivity to changes in feed flow rate (high turndown). As an example, the quality of fluid distribution from a fractal distributor designed for an absorption/distillation process did not

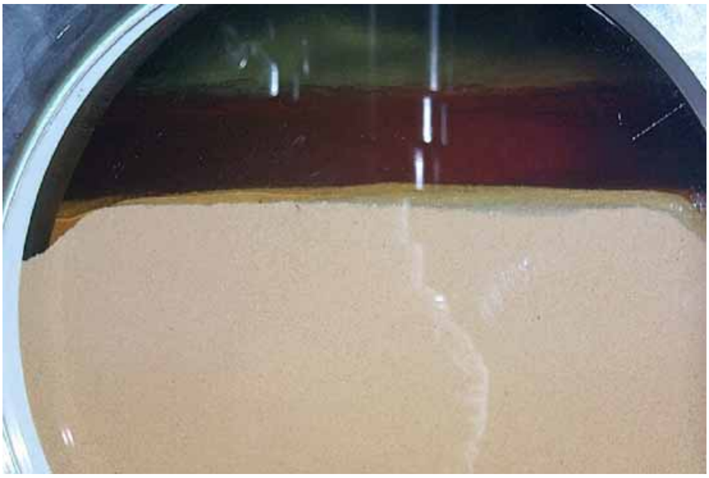

Fig. 5. Interface between water and 15% DS sugar solution above the resin bed.

change within a turndown ratio of 10 *(10)*. The primary reason is that the flow uniformity of fractals is based on symmetries and hydraulic equivalence of flow pathways rather than on the pressure drop criteria used for design of most conventional distributors. The typical very low-pressure drop exhibited by fractals provides additional opportunities for energy savings and novel equipment design.

New Concepts—From Retrofitting to Fractal Design

Selection of inlet/outlet point density and channel size within fractal distributors provides flexibility and control over fluid flow characteristics. Increasing the number of points allows narrowing of the distribution of certain parameters of fluid, such as momentum, concentration, particle size, velocity, and so on. A useful application of this design feature for ion exchange is discussed later.

Introduction of liquid through a fractal distributor with high exit density (several hundred outlets per square foot) leads to a reduction of the fluid momentum exiting each outlet. This inturn reduces the scale of turbulence, minimizing axial mixing. Figure 5 is a snapshot of a sight glass on a commercial ion exchanger during the rinse phase. Water moving downwards displaces colored sugar solution from the resin bed at a very high flow rate—500 bed volumes/h. Although the density difference between the two fully miscible water phases is less than 0.05 g/mL, the presence of a uniform fluid interface demonstrates the absence of turbulence in the headspace of the vessel. In addition to uniform plug flow, this feature of fractal distributors is important because turbulent eddies do not disturb the resin bed. In a conventional distributor design a short bed depth would be difficult to use owing to a low density of distribution points and, hence, high-liquid velocity and momentum.

Conventional ion exchangers utilize resin beds that are relatively deep regardless of process kinetics. In many instances increased bed depth is used to compensate for imperfections of the initial fluid distribution. Our experiments show that contrary to expectations, resin does not distribute liquid radially, and poor initial distribution usually leads to an overall reduction in efficiency. Additionally, increasing bed depth results in higher-pressure drop, which in turn limits equipment throughput. Use of fractal distributors allows constructing equipment with very shallow beds (just a few in. deep). In this case special resins with smaller beads or compressible resins can be used. Jensen et al. *(11)* have indicated that the use of smaller resin beads will increase adsorption kinetics significantly. Smaller diameter beads reduce the diffusion path in and out of the bead resulting in faster kinetics. However, engineering challenges related to pressure drop and fluid distribution were anticipated. As a second case, higher diffusion rates are expected in resins having lower crosslinking (hence, higher water content). In both cases pressure drop across the resin bed will limit equipment throughput. High quality liquid distribution is crucial for improving resin utilization by reducing the chance of premature breakthrough. Fractal equipment provides new opportunities for using these difficult media and for reducing resin inventories.

Applications of Fractal-Based Technologies

Several examples of fractal-based technologies that have found commercial application along with a few technologies under development are discussed later. The results illustrate that dramatic improvements can be made when equipment is designed specifically to take advantage of fractal characteristics.

Juice Softening in the Sugar Beet Industry

Industrial implementation of a new generation of fractal ion exchangers with shallow resin beds has opened new opportunities for the softening process in beet sugar factories (Fig. 6). Near ideal fluid distribution allows utilization of very small resin beds resulting in low capital and operating expenses *(9,12)*. The fractal softener is quite compact (dimensions are 1.5 x 1.5 x 0.3 m^3); however, it operates at the very high flow rate of 500 bed volumes per hour (BV/h).

(Table 1) illustrates the advantages of fractal weak acid softening systems compared with conventional weak acid softening systems. Regardless of high flow rates the pressure drop through the entire system (including two distributors and the bed of resin) does not exceed 0.25 bars, which allows construction of ion exchange cells without the requirement of pressure-rated vessels. Overhead tanks for gravity feed are sufficient for providing feed flow to the cells.

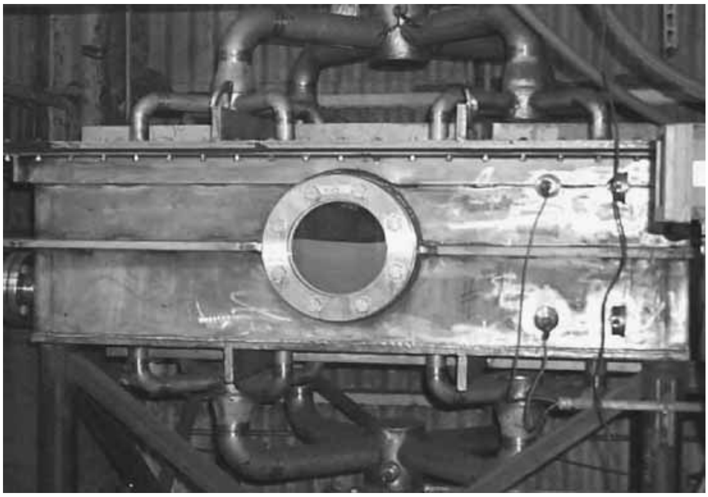

Fig. 6. Fractal softener (an ion exchange vessel with fractal distributors installed on the top and bottom).

Table 1
Comparison of Conventional Weak Cation Juice Softening

Parameter	Conventional (lateral orifice distributor)	Fractal flat bed
Resin bed depth (m)	1.0	0.15
Exhaustion flow rate (BV/h)	50	500
Bed pressure drop (bar)	3.5–5.6	0.1
Regeneration flow rate (BV/h)	30	150

As a result of the introduction of this particular fractal implementation, overall capital cost of the weak cation juice softening process has been reduced to 35–40% of the conventional technology. Energy requirements for pumping have been reduced significantly. Peripheral tanks are smaller in size, which has had a positive influence on installation cost. A similar approach is being evaluated for adsorptive decolorization of sugar solutions. In the initial phase of an ongoing project a threefold reduction of resin bed depth has been demonstrated.

Molasses Desugarization

Molasses desugarization is a process, which has been used commercially in the beet sugar industry for the last 20 yr. The process is based on SMB chromatography and is principally similar to the SMB process used in corn wet milling for fructose purification. Being a final product of sugar processing, molasses still contains about 50% sucrose (based on dry substance), significant amounts of inorganic salts, amino acids, other sugars, and so on. Because of the large variation in molecular weight, size, and affinity of molasses components, separation of sucrose in a relatively pure form presents a challenge. Recovery of 92% pure sucrose with 90% yield has been long considered an industry standard. In a conventional system, an increase in molasses loading per unit of resin typically causes a significant reduction in performance. Introduction of fractal distributors coupled with the use of smaller resin has allowed a reduction of the inventory of ion exchange resin used as separation media by a factor of two without any loss of performance.

Arkenol Process

The potential of fractal technology can be further illustrated with reference to an application in the Arkenol process *(13)*. In this process concentrated sulfuric acid is used to pretreat biomass. The task of acid recovery can be solved using SMB chromatography. A feasibility study performed by Arkenol indicated that acid recovery by chromatography should exceed 98%, and the recovery of sugars in the product fraction should be higher than 98%. Additionally, eluent use should be minimized to reduce dilution (and subsequent energy use for acid reconcentration). A project, in part addressing this problem, was financed by DOE grant no. DE-FC36-01ID-14016. A key accomplishment of the project was the reduction of SMB size by 75% with a simultaneous decrease of product dilution while maintaining required purities and recoveries *(14)*. A short bed design allowed the use of small bead compressible separation media and thus, an improvement in process kinetics. The results would have been difficult (even if possible) with the use of conventional equipment. Japan Gas Corporation (Japan) is presently testing Arkenol technology using fractal SMB systems supplied by Amalgamated Research Inc. (Twin Falls, ID).

Fractal SMB Chromatography on a Model Biomass Conversion Stream

In the initial phase of the above DOE—funded research raw beet juice was considered to be a good model stream for biomass hydrolysate in regards to separation of sugars from impurities. Beet juice is derived from plant material and has many components; some of them poorly identified. The separation task was mainly isolating the groups of components from each other. Despite a high solids loading, it was possible to reduce the SMB system size by a factor of two compared to conventional equipment. The finding has had practical value for a new SMB separation process developed for the sugar industry.

The results of process intensification using fractal technology are summarized in Table 2. The size reduction factor is calculated by dividing the resin volume used in a conventional system by the resin volume in the

Table 2
Process Intensification With Fractals

Technology	Process	System size reduction factor
Juice softening (fast kinetics)	Ion exchange	10
Adsorptive decolorization (slow kinetics)	Ion exchange	3
Molasses desugarization	SMB chromatography	2
Acid recycle from biomass hydrolysate (Arkenol)	SMB chromatography	3
Model biomass stream (high solids loading)	SMB chromatography	2

fractal-based system. The equipment size reductions are quite impressive and may be improved further with more efficient separation media. Another way to present the system size reduction factors is to use them as multipliers for calculating the reduction in energy use and increase of specific loading per unit of separation media.

Existing biorefinery applications are not listed in Table 2 (e.g., HFCS production) may also benefit from fractal technology either via retrofitting or with new equipment.

Conclusions

It has been demonstrated that fractal-based technologies are used effectively in existing biorefineries. The equipment size and energy requirements can be significantly reduced owing to improvement of process efficiencies. Risk factors owing to implementation of fractal technology on a large scale are reduced dramatically because of the inherent scalability of fractal-based equipment. A high level of collaboration is needed to validate fractal concepts applied to process streams expected from future biorefineries. Manufacturers of resins and other separation media should consider the benefits of providing materials that take advantage of the possible synergistic relationship between media and fractal-based equipment.

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