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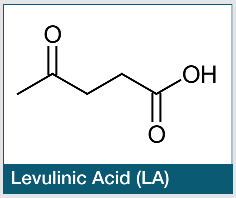
CONVERSION OF BIOMASS TO LEVULINIC ACID, A NEW FEEDSTOCK FOR THE CHEMICAL INDUSTRY

Levulinic acid has very recently received increasing attention as platform chemical for the production of bio-based strategic derivatives. This paper highlights the long walk, not yet completed, to arrive at the industrial production of this biomass derivative and its important applications.

The catalytic conversion of renewable resources into chemical intermediates or biofuels represents a sustainable and environmental friendly solution to their increasing demand. This strategy allows security of supply and economical advantage, in particular when cheap or, indeed, waste or residue raw materials, having a negative value, are employed as starting materials [1-2]. This last aspect is very important because the employment of waste renewables for chemical transformations does not compete with food and feed production in the well known "food vs. fuel (or chemicals) conflict" [3].

In this context levulinic acid (LA) represents a platform chemical which was identified as one of the most important sugar based building blocks from lignocellulosic biomass by National Renewable Energy Laboratory (NREL) and PNNL (Pacific Northwest National Laboratory) researchers [4]. They identified twelve chemicals building blocks that can be produced from sugars via biological or chemical conversions which can be subsequently converted to a number of high-value bio-based chemicals or materials. The final selection of 12 building blocks began with a list of more than 300 candidates: levulinic acid was defined a versatile intermediate and one of the United States Department Energy's (DOE's) top 12 derivatives feedstocks [5]. Levulinic acid (LA), also known as 4-oxopentanoic acid or γ-ketovaleric acid, is a very important platform for chemical industry. It is a C_5 fatty acid, with a ketone carbonyl group and an acidic carboxyl group.



Levulinic Acid (LA)

The presence of both these groups results in interesting reactivity patterns. Since LA is an highly polar compound it is easily soluble in water, ethanol, diethyl ether, acetone and many other organic solvents and it is a versatile chemical intermediate.

In spite of its great potential as a platform chemical, up to now LA has not been produced in a significant volume for large scale distribution and industrial applications. The reason is that LA production was mainly studied in the early 1950s, when expensive precursors such as sucrose or glucose were proposed and the yields were low because of the high amount of by-products. In this context, now the LA synthesis by controlled acid-catalyzed degradation of biomass derivatives appears very promising for an industrial application.

On a molecular level, the conversion of lignocellulosic biomass to levulinic acid is known to follow a complicated reaction scheme involving several intermediates and by-products (see Fig. 1) [6]. Hemicellulose and cellulose, two of the three main constituents of biomass, are carbohydrate-based polymers that can be broken down to low molecular weight sugars by hydrolysis using an acid catalyst. The acid-catalysed decomposition of the C_6-sugar fragments (e.g., glucose) leads to 5-hydroxymethyl-2-furaldehyde as an intermediate product, which is subsequently re-hydrated to give levulinic and formic acids as final products. Hydrolysis of the C_5-sugars of hemicellulose may lead to furfural. In addition, other constituents of the hemicellulose components may produce side products like acetic acid and galacturonic acid. Lignin, the third main constituent of lignocellulosic biomass, is a resin-like polymer matrix with various substituted phenolic groups present.

Generally biomass consists of 38-50% of cellulose, 23-32% of hemicellulose and 15-25% of lignin, the chemical composition of biomass strongly depending on its source.

The simplified reaction scheme given in Fig. 1 does not show the reactions leading to the undesired insoluble-polymeric materials known as humins. LA can be obtained by acid degradation of hexoses, or of any carbohydrate containing hexoses, with co-production of formic acid.

Batch processes for levulinic acid production

Many starting materials such as glucose, sucrose, starch, cellulose and biomass materials as dedicated crops, wood sawdust, cane sugar, wheat straw, and agricultural wastes (e.g. tobacco chops, rice hulls), waste material with cellulose high content (e.g. old newspapers, paper mill sludge…) or sugar high content (e.g. sugar cane, grape or olive mill sludge) have been used to produce LA.

Two different approaches are commonly applied for the hexoses acid catalyzed thermal degradation to levulinic acid:

1) mineral acids in high concentration (e.g., 15-16 N HCl or 31-70 wt% H_2SO_4) as catalysts and low operating temperatures (20-50 °C) [7, 8];
2) highly diluted acids at high operating temperatures (170-240 °C). This is the most promising approach for an industrial application and research studies applying this approach are abundant [6, 9-11].

The big disadvantages of the first approach are the high costs for the strong acid recycle, for the reactor material, and for the product recovery system. On the contrary, hydrolysis with diluted acids is preferred and does not require high consumption of catalyst.

The maximum theoretical yield of LA from hexoses and cellulose is 64.5 wt% and 71.5 wt% respectively, due to formic acid co-production. As close to this theoretical yield in the conversion process could be achieved, it will depend on the extent of the degradation reactions involved.

The yield is generally lowered (two third or even less) by the formation of undesired solid black by-products, called humins, which can cause

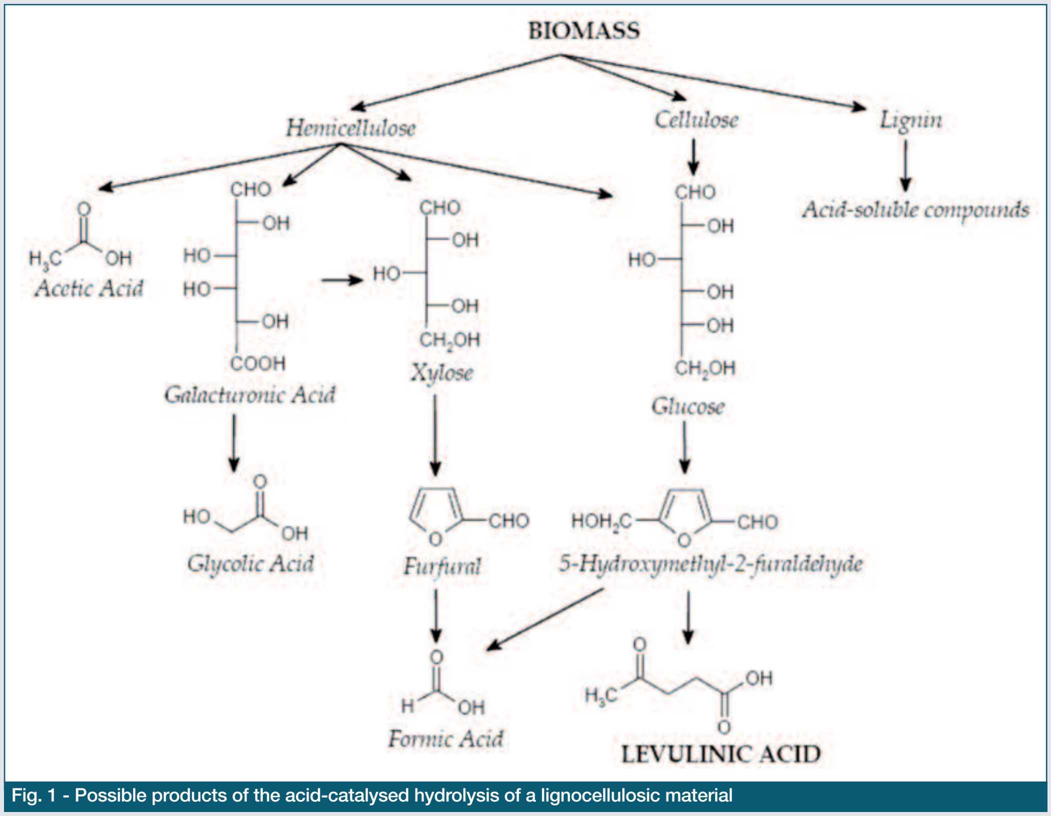


Fig. 1 - Possible products of the acid-catalysed hydrolysis of a lignocellulosic material

Tab. 1 - Batch processes for the biomass conversion to LA

Biomass	C_f[a]	Acid	C_{acid} (wt%)	T (C°)	time (h)	Y_{LA}[b] (wt%)	Ref.
Cane sugar	28	HCl	18	100	24	15	13
Glucose	29	HCl	6,5	162	1	24	14
Fructose	29	HCl	6,5	162	1	25	15
Starch	26,5	HCl	5,2	R.T.	24	19	16
Rice hulls	14	HCl	1	160	3	10,3	16
Com Stalk	14	HCl	1	160	3	7,5	17
Sucrose	27	Amberlite IR-120	19	R.T.	41	15,6	18
Fructose	27	Amberlite IR-120	19	R.T.	27	23,5	19
Glucose	27	Amberlite IR-120	19	R.T.	124	5,8	20
Pulp slurry	10	HCl	6	160	1	40,5	21
Glucose	10	HCl	6	160	0,25	41,4	22
Wood sawdust	20	HCl	1,5	190	0,5	9	23
Bagasse	9	H_2SO_4	1,3	25-195	2	17,5	24
Fructose	4,5-18	HCl	2-7,5	100	24	52	25
Sucrose	20	Resin Dowex	6,25	100	24	17	26
Fructose	50	LZY-zeolite	50	160	15	43,2	27
Glucose	12	HY-zeolite	3	150	24	6	28
Cellulose	10	HBr	1-5	150-250	2-7	≤26,9	29
Aspen wood	10	H_2SO_4	1-5	150-250	2-7	≤15,5	29
Newspaper sludge	30	H_2SO_4	10	150	8	12,8	30
Sorghum grain	10	H_2SO_4	8	200	0,67	32,6	31
Extruded starch	25	H_2SO_4	4	200	0,67	47,5	32
Wheat straw	6,4	H_2SO_4	3,5	209,3	0,63	19,8	33
Water hyacinth	1-5	H_2SO_4	0,1-10	150-175	0,33	9	34
Sucrose	16	Nafion-NR50	7	140	40	35	35
Marine algae	4,7	H_2SO_4	3	160	0,67	19,5	36
Rice straw	10	H_2SO_4	6	220	0,6	28,4	37
Cellulose	10	Sulphated TiO_2	7	240	0,25	27,2	38

[a] C_f is the initial concentration of feedstock and defined as the ratio between the mass of feedstock and the total mass

[b] Y_{LA} is defined as the ratio between the mass of LA and the mass of feedstock

[c] R.T. = Reflux Temperature

the clogging of the reactor and separators and filters fouling [12]. Furthermore, humins deposition on reactor walls makes the thermal exchange and the process temperature control difficult, with consequent further decrease of selectivity.

Many kinds of catalysts have been used for these batch processes: homogeneous (mineral acids like HCl, H_2SO_4, HBr) and heterogeneous ones (acid resins, zeolites, acid oxides…) (Tab. 1) [13-38].

The first experiments on biomass conversion to levulinic acid in an application perspective has been patented by Moyer in 1942. Starch and hydrochloric acid were used as raw material and catalyst respectively. The reaction slurry was converted in autoclave operating at 200 °C with a yield of 35 wt% and high formation of huminic by-products [16]: a relatively long time was required to complete the reaction. When waste materials were utilized as feedstock, yields were lower. Sassenrath and co-workers employed wood sawdust with dilute HCl at 190 °C and reported a very low ponderal yield of levulinic acid: 9 wt% [23]. Recently, a patent has been reported the production of levulinic acid from old newspapers, a feedstock with very high cellulose content: 85 wt% [30]. In this process there is a first pre-hydrolysis step with sulphuric acid at 40 °C to obtain the cellulose de-crystallization. Successively the reaction mixture is heated up to 150 °C for a long reaction time (8 hours). The levulinic acid ponderal yield is very low (12.8 wt%), therefore this patent appears not promising because the reaction time is high but the yield low for a starting material with high cellulose content.

In 2002, Fang studied the LA production from sorghum grain (starch content 74%) with sulphuric acid at 200 °C [31]. In this work H_2SO_4 resulted better than HCl. The obtained yield is quite high (33 wt%) but the feedstock is not a waste material and sulphuric acid is employed in high concentration (10 wt%), therefore this process can be considered expensive for an industrial application.

More recently Chang reported the conversion of milled wheat straw as starting material with a cellulose content of 40.4% [33]. The optimal conditions, which allow to reach a yield of 19.8 wt%, about 70% of the maximum theoretical yields based on cellulose content, involved the tight control of the reaction temperature at 209 °C.

In 2008, a research group of the University of Groningen reported the study on acid catalysed hydrolysis of the water hyacinth plant (very diffused in Holland, but invasive for Italy) to levulinic acid [34]. With high sulphuric acid concentration, LA was the major compound formed, with a yield of 53% in mol (based on dried C_6 sugars) and 9% in weight. In this last year, a very interesting work was proposed by the group of Jeong, who studied the catalytic conversion of the marine algal biomass *Gelidium amansii* to sugars (glucose and galactose) and levulinic acid [36]. This red alga is widely used in agar production, applied in food and chemical industries, and has a high carbohydrate content. The main disadvantage of the use of heterogeneous acid catalysts is represented by their deactivation by tar, humic substances formed in the quantity, at least, of 5-10 wt% based on the target products. The other problem with resins and zeolites is their generally low catalytic activity, so higher amount of acid is necessary to obtain good LA yields. We can compare the catalytic activity of homogeneous and heterogeneous catalysts using fructose as substrate. When HCl is employed the yield is 52 wt% working at low acid concentration and low process temperature (100 °C) [25]. On the contrary, using the heterogeneous catalyst LZY zeolite, to obtain a LA yield of 43 wt% is necessary higher temperature (160 °C) and high catalyst amount [27].

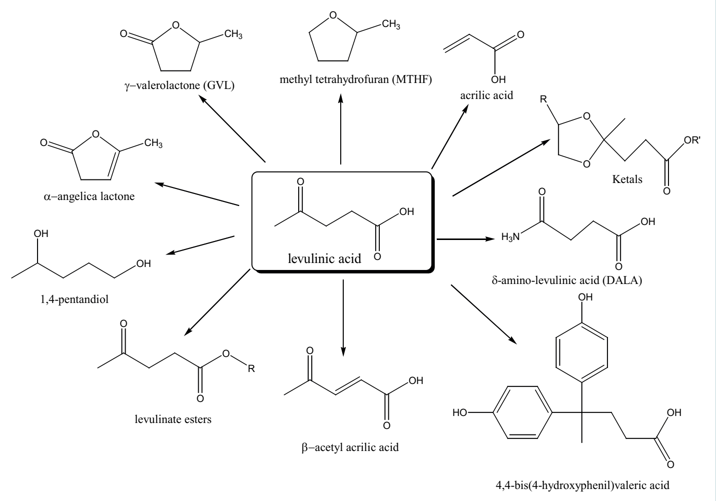


Fig. 2 - Potentially interesting derivatives of levulinic acid

Continuous processes for levulinic acid production

All the above described processes were carried out in laboratory scale using batch reactors. Dunlop and Wells, in 1957, proposed the first continuous process for the production of LA from furfural residue working at atmospheric pressure [39]. The acid catalyst used in this process was dilute sulphuric acid (3 wt%) and the biomass adopted was corncob furfural residue (21 wt%): all the components are premixed with water and then reacted working at 170 °C for two hours. After this reaction time, the solid humins were separated by filtration and the mixture was extracted with methyl isobutyl ketone to obtain an organic solution containing LA and an aqueous phase with the acid catalyst. The latter is recycled to the mixer while the extraction solvent is separated from LA in an evaporator and then recycled to the extraction column. Further concentration and purification of LA was carried out in a fractionation unit by vacuum distillation. A LA yield of 19.9 wt% based on the weight of the dry feedstock was claimed.

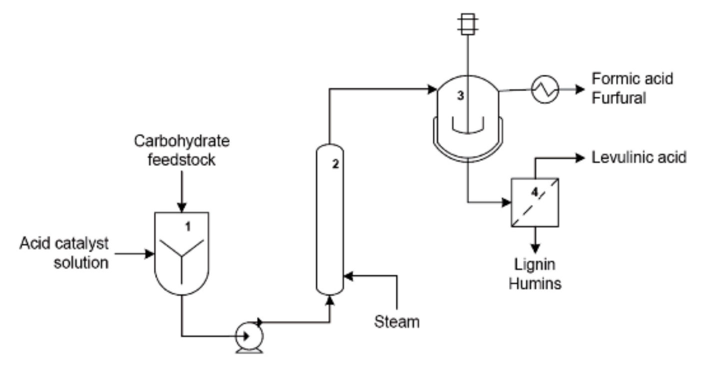
A very different approach to a continuous process was patented by Ghorpade *et al.* in 1999 [40]. They studied the reactive extrusion of corn starch, sulphuric acid and water: the reactants were premixed and then sent to a twin-screw extruder with a variable temperature profile from 80 to 150 °C. The solid humins were separated by filtration and LA collected by vacuum distillation: the yield was 48% on the weight of the feedstock. The main disadvantage of this process is the need of a very expensive special steel extrusor because of the high corrosiveness of reaction mixture and the very high energy consumption.

The Biofine Process, proposed by Fitzpatrick, has represented one of the most celebrated technologies currently claimed [41]. This process involves the use of dilute sulphuric acid as a catalyst and many feedstocks (such as paper, wood and other ligno-cellulosic material) have been claimed as starting materials, working with two different hydrolysis reactors to give LA and furfural as the final products.

The feedstock is initially shredded, then the biomass particulates are conveyed by a high-pressure air injection system to a mixing tank. Here the feedstock is mixed with recycled dilute sulphuric acid (1.5-3%, depending on feedstock). The Biofine Process (Scheme 1) then consists of two distinct acid-catalyzed steps. In the first step biomass and sulphuric acid solution are mixed, and continuously supplied to a tubular reactor that operates at a typical temperature of 210-220 °C and pressure of 25 bar. The residence time in this first reactor is only 12 seconds, in order to depolymerise the polysaccharides into their soluble monomers (e.g. hexoses, pentoses, HMF).

According to the patents claims, a small amount of sodium (or potassium) sulphite is added to the reaction mixture in order to prevent the clogging of this reactor, characterized by an elevated height/internal diameter ratio. However, up to now, it is not well clear the role of this component in order to prevent humins deposition. The outflow mixture is fed to a continuously stirred tank reactor that operates at a lower temperature (190-200 °C, pressure 14 bar). This reactor is considerably larger than the first one and a residence time of approximately 20 minutes is adopted. In this second step the complete hydrolysis to levulinic acid takes place. The reaction conditions in the second reactor are chosen as such as to vaporise formic acid and furfural, and the vapour is externally condensed to collect these side products. Furfural and other volatile products tend to be removed at this stage whereas the tarry mixture of LA and residues are passed to a gravity separator. From here, the insoluble mixture goes to a dehydration unit where the water and volatiles are boiled off. The heating of the mixture to boil off LA is carried out under reduced pressure and results in the tarry material being “cracked”, to give a powdery substance (‘char’). Solid by-products are removed from the LA solution in a filter-press unit and the crude 75% LA product can be purified up to a purity of 98% by distillation. The acid is recovered in the final recycle stage, allowing it to be reused in the system.

The Biofine Process claims from cellulose LA yields of 70-80% of the theoretical maximum value. This translates to the conversion of approximately 50% of the mass of 6-carbon sugars to LA, with 20% being converted to formic acid and 30% to tars. The mass yield of furfural from 5-carbon sugars is also approximately 70% of the theoretical value of 72.7%, equivalent to 50% of the mass, the remainder



Scheme 1 - Scheme of the Biofine continuous process
(1: premixer; 2: tubular reactor; 3: stirred reactor; 4: filter-press)

being incorporated in the char. The yield claimed is 62% by employing wastepaper such starting material. This material contains 80 wt% of cellulose so it cannot be defined a real waste feedstock. Surely, by employing two different special steel reactors, the process costs are very high. To the best of our knowledge, the first commercial-scale plant for the conversion of lignocellulosic biomass to LA has been built in Caserta, Italy [42-44]. At first, this plant adopted the Biofine technology working with the two reactors configuration using local tobacco chops or paper mill sludge as feedstock.

The process with tobacco chops evidenced many disadvantages of the Biofine approach, in particular due to the dramatic complete clogging of the small diameter first reactor by salts and humins deposition. Thus, University of Pisa was asked to study the process improvement. Our investigation evidenced that very diluted HCl always resulted a better catalyst in comparison with H_2SO_4, adopting only a continuously stirred tank reactor. In fact, in the presence of H_2SO_4, significant deposition of solid humins by-products on the reactor walls was observed. Moreover, due to the significant content of calcium salts in tobacco chops (calcium concentration >4 wt.%) significant deposition of calcium sulphate and reactor clogging took place.

On the contrary, under our optimized reaction conditions the complete absence of humins deposition was ascertained. The optimum conditions for LA synthesis were different for each raw material and a pre-hydrolysis step at a lower temperature (<100 °C) can help to increase the yield. Also the addition of a cheap electrolyte such as NaCl increased the yield for some substrates. It is in fact known that the addition of an electrolyte increases the rate of the heterogeneous acid hydrolysis of the difficulty accessible portion of cellulose to give glucose and then LA [45]. When a water slurry of tobacco chops powder, containing 25 wt.% of cellulose, was employed as starting material, the actual yield of LA of about 15 wt.% was reached. This value represents the 84% of the theoretical yield, the highest up to now never reported using a raw biomass [46].

At the end of the reaction a solid residue, retaining many of the functionalities of lignin, was always recovered, its amount depending on the acid-insoluble lignin content in the starting biomass. Indeed the economics of the whole industrial process are also related to its exploitation for many applications: as soil conditioner, as free radical scavenger to be used as an antioxidant, as low-density filler instead of inorganic fillers, as a component for biodegradable composits formulations. As a curiosity, we studied also the conversion to LA of the exhausted lemon peels deriving from "Limoncello" liqueur production: they represent a significant waste material for Campania Region (about 15 lemons are necessary for any liter of pure alcohol and about 3×10^6 liters of Limoncello are produced in Campania). The lemon rind indeed contains cellulose and sugars that could be hydrolyzed to LA. Under optimized conditions 22 weight percent of the feedstock was converted into LA without humins by-products deposition.

Levulinic acid: a versatile intermediate

LA is a platform chemical with various potential uses: it can be used as solvent, antifreeze and as starting material for polymers and pharmaceutical compounds. The presence of two functional groups makes levulinic acid a potentially versatile building block for the synthesis of strategic bulk chemicals as shown in Fig. 2 [47-48].

In particular, various levulinate esters may be used as gasoline and biodiesel additives. δ-Aminolevulinate (DALA) is a known herbicide, and the bisphenol derivative may be an interesting substitute for bisphenol A in polymer synthesis [49]. A very important LA derivative, obtained from its selective hydrogenation, is γ-valerolactone (GVL) [50]. It exhibits very attractive physical and chemical properties and could be considered as a sustainable liquid [51]. It can be used in perfumes and food industries as well as in polymer synthesis. 2-Methyltetrahydrofuran (MTHF) is a potential fuel additive obtainable from GVL catalytic hydrogenation [52]. In this last year another very important class of LA derivatives, levulinic ester ketals, has received an enormous interest in the field of bio-based derivatives, in particular in USA, where the biomaterials company Segetis has patented these novel compounds [53]. These ketals are formed from the reaction of a levulinic ester with a polyalcohol, typically glycerol, to form the ketal chemical moiety. Levulinic ester ketals show broad solvency and compatibility and good thermal stability. Currently, their main applications

under development are in the field of plasticisers (a potential outcome could be the replacement of phthalate plasticizers with these biobased derivatives), solvents, thermoplastics, and polyurethanes.

Conclusions

The use of waste renewable feedstocks for the industrial production of levulinic acid can provide a valuable route to the generation of very important biobased chemicals. The hydrolysis reactions were carried out in a broad range of reaction conditions in batch and in continuous processes, at different temperatures, with different types of acid catalysts and concentrations and initial biomass intakes. It has been demonstrated that the yield and the selectivity to levulinic acid strongly depend on the applied reaction conditions, which must be optimized for each biomass type, considering the matrix and the metal ions which are present. The optimization of the reaction parameters and of the type of reactor has permitted to reach high levulinic acid yields with a very low by-products (humins) formation. Further process improvements by using new technologies (such as microwave irradiation instead of traditional heating) and new raw materials (such as non food crops) are in progress in our research group.

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RIASSUNTO

Conversione di biomassa in acido levulinico, un nuovo prodotto di partenza per l'industria chimica

L'acido levulinico ha focalizzato negli ultimi anni un crescente interesse come platform chemical per la produzione di bioderivati di interesse strategico. Il presente lavoro mette in evidenza il lungo percorso, non ancora completato, per arrivare alla produzione industriale di questo derivato della biomassa e le sue importanti applicazioni.