Renewable sources are the key for a sustainable energy supply model. Within this context, ethanol is emerging as an efficient and economically viable liquid biofuel. However, there is a lot of controversy about the competitive use of food commodities as biofuel sources. To solve this problem lignocellulosic raw materials have been considered as non-edible sources of fermentable sugars, but the technologies required for large-scale conversion of lignocellulosic biomass into biofuels are not yet fully developed. The clean and rapid technology of sub/supercritical water hydrolysis is proving to be an attractive alternative to conventional chemical and biological processes. However, further optimization of operation conditions (temperature, time, solvent: solid proportion) is still needed so that it can be scaled-up to industrial level. A semi-batch equipment was used to perform subcritical water hydrolysis of four residues of food industry: sugarcane bagasse; grape seed previously defatted by supercritical fluid extraction; pressed palm fiber; coconut husk. The hydrolysis process was conducted at 208 °C and 20 MPa; total reaction time was 32 min. Reducing sugars and total reducing sugars were determined by a colorimetric method. The sugars recovery rate increased up to a maximum and then decreased for all the raw materials. For sugarcane bagasse hydrolysis the liquefaction degree was 68 %, reducing sugars recovered were 7 % and total reducing sugars were 15 %, implying that there were mostly oligosaccharides in the hydrolysate.